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NO. 232

"Development and Application of a Scanning Ion Microprobe"





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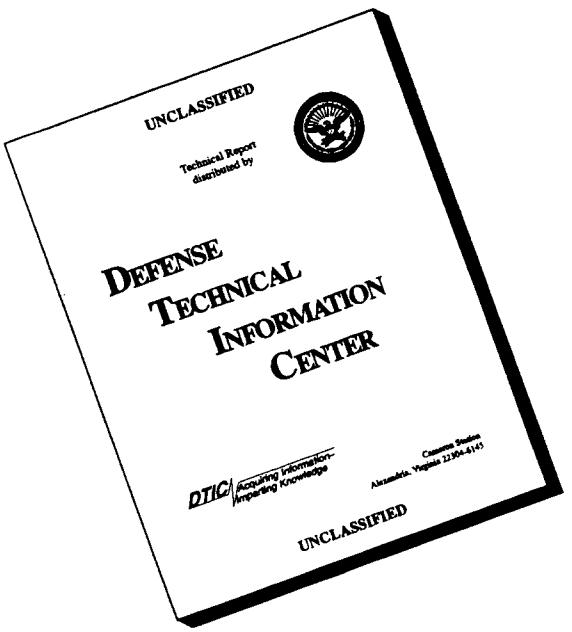


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13. ABSTRACT (Maximum 200 words)

A complete scanning ion microprobe system was developed for use in the Naval Academy Tandem Accelerator Laboratory. The microprobe employs a computer-controlled positioner to scan a sample while it is bombarded with a finely focused particle beam. The beam excites characteristic X-rays from the sample atoms. The X-ray yields are converted to elemental concentrations. The concentration and position data are then used to create two dimensional surface concentration maps, which are analogous to microscope views of the sample. The microprobe was tested using a 200-mesh transmission electron microscope grid. The diameter of the beam spot on the sample surface was found to be approximately 30 $\mu$m. The capabilities of the microprobe were demonstrated by measuring the positional variation of elemental concentrations in several inclusions of the Allende meteorite. These inclusions are thought to predate the formation of the solar system. Extensive position dependent concentration data may eventually provide information on the formation mechanisms and temperature and radiation history of meteorites.

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# "Development and Application of a Scanning Ion Microprobe"

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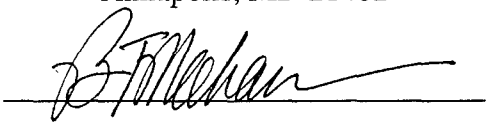

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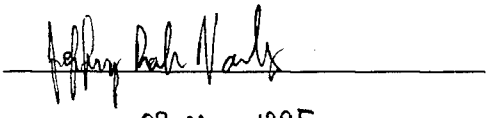

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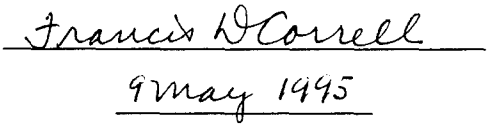

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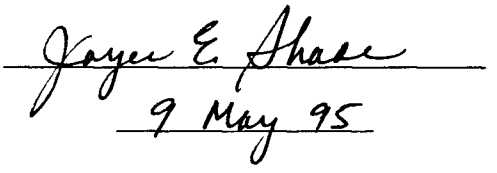

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# "Development and Application of a Scanning Ion Microprobe"

Abstract

A complete scanning ion microprobe system was developed for use in the Naval Academy Tandem Accelerator Laboratory. The microprobe employs a computer-controlled positioner to scan a sample while it is bombarded with a finely focused particle beam. The beam excites characteristic X rays from the sample atoms. The X-ray yields are converted to elemental concentrations. The concentration and position data are then used to create two dimensional surface concentration maps, which are analogous to microscope views of the sample.

The microprobe was tested using a 200-mesh transmission electron microscope grid. The diameter of the beam spot on the sample surface was found to be approximately 30 μm.

The capabilities of the microprobe were demonstrated by measuring the positional variation of elemental concentrations in several inclusions of the Allende meteorite. These inclusions are thought to predate the formation of the solar system. Extensive position dependent concentration data may eventually provide information on the formation mechanisms and temperature and radiation history of meteorites.

KEYWORDS: Scanning Ion Microprobe, Ion-Beam Analysis, PIXE, Meteorites, Carbonaceous Chondrites

TABLE OF CONTENTS

# I. Introduction

The most popular methods for determining the elemental composition of a material usually employ chemical analysis techniques. These techniques have very good sensitivity to minute concentrations of the elements, but they are time consuming and require much sample handling. Furthermore, the sample is frequently destroyed in the process, which is unacceptable in the case of rare items or materials requiring subsequent analysis by other methods. The popularity of accelerator-based techniques using proton-induced reactions is growing rapidly because these techniques are not only sensitive and accurate, but they usually also leave samples undamaged.

Proton Induced X-ray Emission (PIXE) Spectroscopy is one of several Ion Beam Analysis (IBA) techniques employed at the Naval Academy Tandem Accelerator Laboratory (NATALY). Figure 1 demonstrates this technique. An energetic beam of protons from an accelerator disrupts the electronic configuration of atoms in the sample. As the electrons return to the ground state configuration, X rays are emitted. Because each element in the Periodic Table has a unique electronic configuration, each element produces a characteristic set of X rays. A representative X-ray spectrum (in this case, of the Allende meteorite) is shown in Figure 2. The energies of the peaks identify elements present in the sample, while the intensities reflect the concentrations. In practice, PIXE works well for elements with atomic numbers between sodium (atomic number 11, $K_\alpha$ X-ray energy = 1.041 keV) and tin (atomic number 50, $K_\alpha$ X-ray energy = 29.193 keV) [Jo88].

If the sample to be studied is spatially homogeneous, the size of the ion beam is unimportant so long as it is smaller than the sample. Most of the research at NATALY has involved such homogeneous samples [Ma91], and their analysis has been performed using proton beams with diameters between 1 and 2 mm.

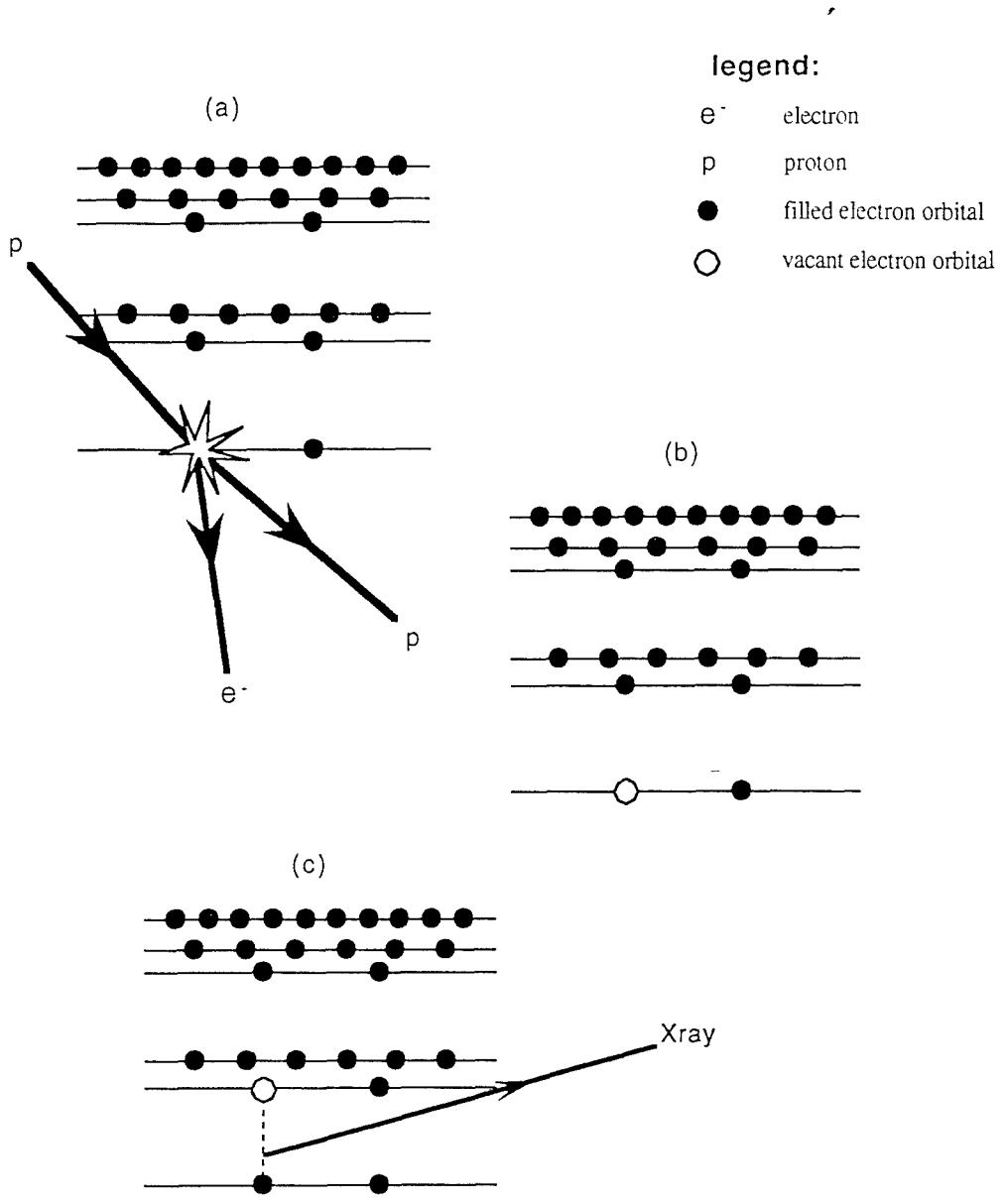


Figure 1. (a) An incident proton ejects an electron, indicated by a filled circle, from a low-lying energy level of an atom. (b) A vacancy remains, indicated by an open circle. (c) An electron from a higher energy level fills the vacancy, emitting an X ray.

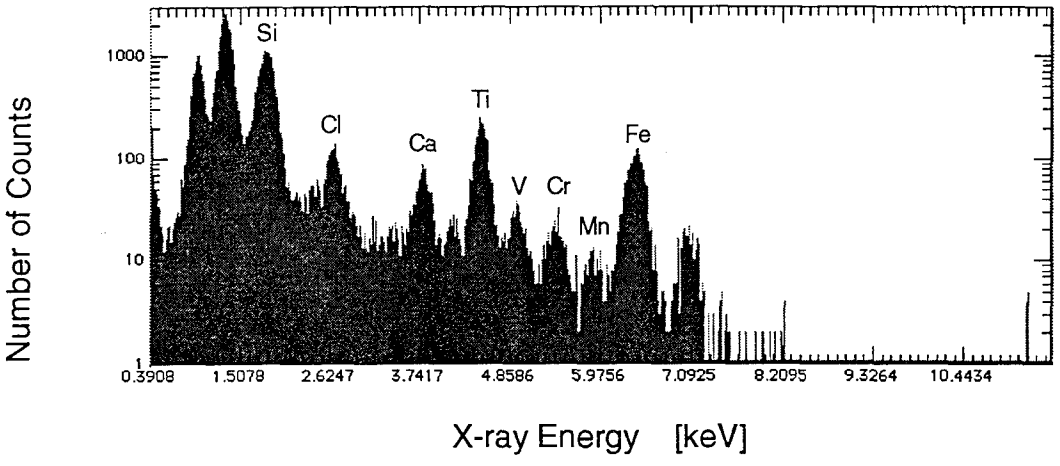


Figure 2. A typical PIXE spectrum obtained from the Allende meteorite. The histogram displays the number of X rays counted versus the X-ray energy. Peaks corresponding to several elements are labeled.

Some materials of potential interest, such as the annual growth rings of trees or oyster shells, have compositions that vary significantly with position. To study samples of this kind, a system that produces intense particle beams of small diameter, in some cases as small as a few μm, is required. Such systems are commonly known as ion microprobes. If either the particle beam or the sample can be moved or scanned in a systematic way, so that one or two dimensional maps can be made of elemental concentration versus position, the system is known as a scanning ion microprobe.

Although small accelerators suitable for IBA are common, scanning ion microprobe systems are still fairly rare. Recent literature reviews [Lo93], [Do91], [Tr90] describe fewer than 10 scanning ion microprobes in the United States and fewer than 50 worldwide. They are used to study minerals, biological materials, archaeological artifacts, and a wide variety of other samples.

State-of-the-art microprobes can produce particle beams as small as 1 μm in diameter, but these are expensive, bulky, and temperamental. In early 1994, the Naval Academy acquired equipment to construct a simple scanning microprobe that would produce beams with diameters on the order of 50 μm or less. To make a complete microprobe system, this equipment needed to be integrated with specially developed computer hardware and software for controlling the sample position and handling the data.

This project developed and tested such a system. In its final form, the microprobe can create one or two dimensional maps of the elemental concentration on the surface of a sample. Such measurements involve gathering and managing several hundred spectra from various positions on the sample and coordinating the data acquisition and sample positioning processes. A highly-automated data acquisition procedure was developed to handle the large number of operations that must be performed.

Chapter II describes the equipment and discusses the design and use of data acquisition and analysis packages. Chapter III reports on performance testing of the scanning microprobe using a transmission electron microscope grid. Chapter IV illustrates typical measurements that can be performed with the completed microprobe using the Allende meteorite as an example. Appendix I and Appendix II contain most of the detailed information regarding software and data manipulation procedures.

# II. Equipment, Data Acquisition, and Data Reduction

## II.A. Accelerator and Beamline Layout

The proton beam is produced using the Naval Academy's National Electrostatics Corporation model 5SDH Tandem Electrostatic Accelerator, which is illustrated in Figure 3. This accelerator has a maximum terminal potential of 1.7 MV and can produce proton beams with kinetic energies up to 3.4 MeV.

The proton beam begins in the SNICS II (Source of Negative Ions by Cesium Sputtering) ion source as a beam of negative hydrogen ($H^-$) ions produced by bombarding a titanium hydride pellet with cesium ions. The $H^-$ ions are pre-accelerated to an approximate energy of 15 keV before injection into the tandem accelerator. The pre-acceleration is accomplished by keeping the SNICS source at a large negative potential with respect to the input of the tandem accelerator.

The $H^-$ beam is attracted to a terminal in the middle of the accelerator which is held at a high positive potential. An Einzel lens at the entrance of the accelerator focuses the ions into a "stripper canal" in the terminal. In the stripper canal, the $H^-$ ions are converted into protons when the two electrons on the $H^-$ ion are removed by collisions with low pressure nitrogen gas. The protons then emerge from the stripper canal and are repelled by the positive terminal. Thus, the beam particles are accelerated twice by the same potential: once as negative ions, and once as positive ions. As a consequence, the tandem accelerator design gives singly charged ions twice the energy that they would acquire in a single stage accelerator having the same terminal potential.

As the proton beam leaves the tandem accelerator, it passes through the magnetic quadrupole doublet lens. This lens focuses and shapes the beam in much the same way that an optical lens would shape a beam of light. At NATALY there are two beamlines downstream of the high energy quadrupole. One of these is the microprobe beamline

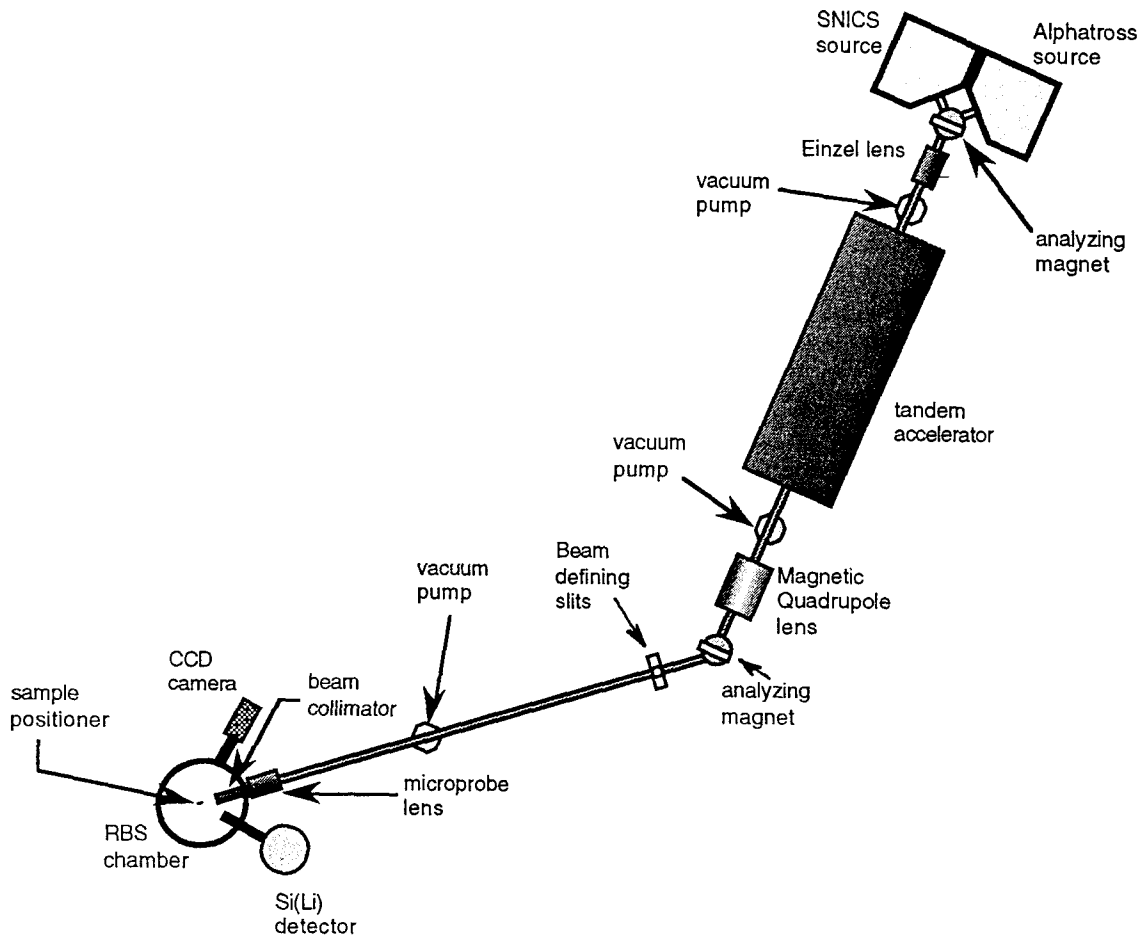


Figure 3. Layout of major equipment in the Naval Academy Tandem Accelerator Laboratory.

used for elemental analysis, and the other is a general-purpose beamline. An analyzing magnet is used to bend the particle beam into the desired beamline.

For microprobe analysis, in which the goal is to probe the fine structure of materials, it is frequently necessary to have particle beams with diameters on the order of μm or tens of μm. At NATALY, these are obtained through the use of beam-size-defining slits, an electrostatic microprobe lens, and a collimator. The high energy magnetic quadrupole doublet and the analyzing magnet focus and steer the beam onto the beam defining slits. The aperture formed by these slits acts as the object for the electrostatic microprobe lens. The microprobe lens [Kl91] is a series of four alternating-polarity electric quadrupoles, a configuration commonly referred to as a "Russian Quadruplet". The lens uses an object distance that is long in comparison to its image distance to reduce the beam diameter by about a factor of 10. With an object aperture of about 0.3 mm, the lens typically produces a 30-μm-diameter beam at the target. A collimator just after the microprobe lens intercepts any stray beam particles.

After being focused by the microprobe lens, the beam enters the Rutherford Backscattering Spectrometry (RBS) chamber shown in Figure 4. The RBS chamber is a versatile component that supports several different measurement techniques. The sample is mounted in the chamber on a 5 axis computer-controlled goniometer which allows it to be moved around in the beam. The chamber also houses mountings for X-ray, gamma-ray, and charged-particle detectors as well as a viewport for a CCD camera so that experiments can be observed on a television monitor.

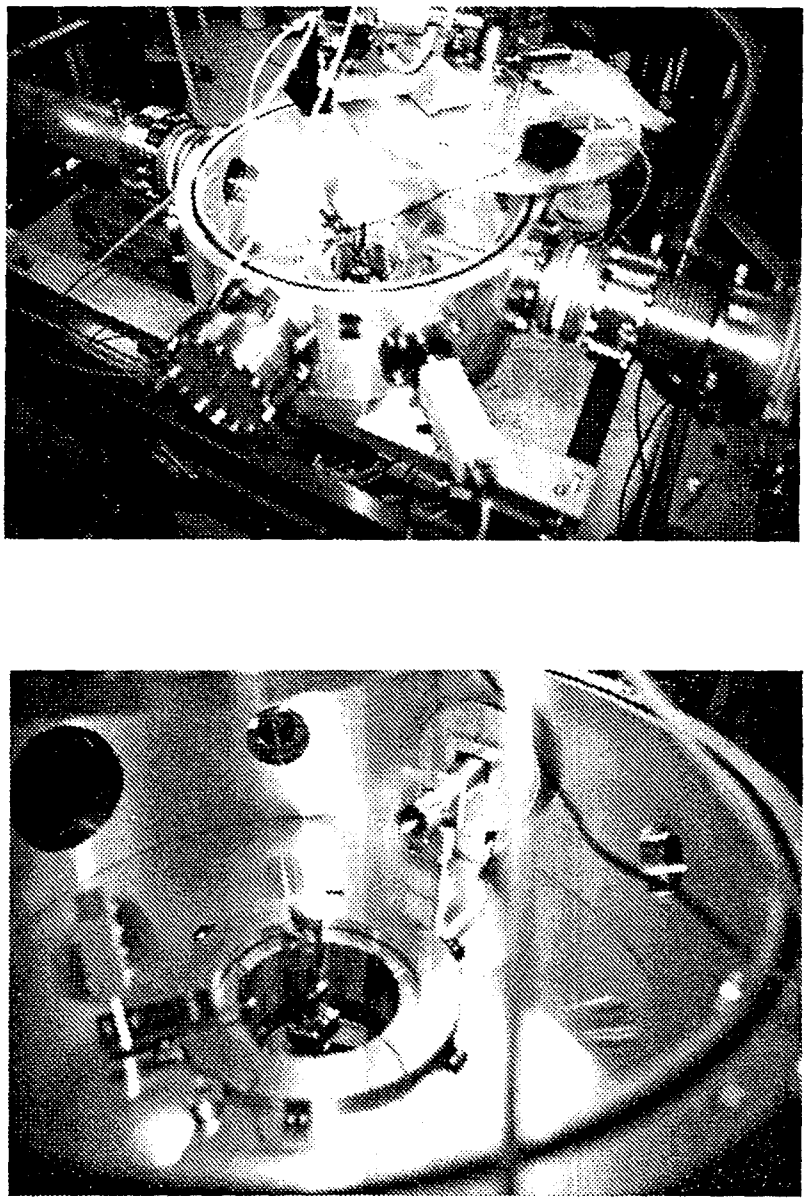

Figure 4. Two views of the RBS chamber. In the upper view, the proton beam enters from the beam pipe on the right. Samples are mounted on a goniometer at the center of the chamber. The CCD camera is visible as a light-colored rectangle in the lower middle of the picture. In the lower view, the inside of the chamber is shown from a different viewpoint. The beam enters through a collimator in the upper middle of the picture, and the goniometer is visible in the center.

## II.B. Data Acquisition Electronics

The programming environment used for data acquisition is a relatively new commercial product for Macintosh computers called Kmax™ [Sp93]. Kmax™ employs the Computer Assisted Measurement and Control (CAMAC) interface commonly used in nuclear science and process control and takes advantage of the natural windowing capabilities of the Macintosh operating system. Kmax™ allows a user to control a very large variety of devices that conform to the CAMAC standard, including Analog to Digital Converters (ADCs) and scalers. Kmax™ also permits on-line sorting of data into histograms and provides a set of functions for simple on-line analysis. Through the use of the Macintosh resource editor ResEdit™, one can incorporate external software drivers (XCMDs), such as those used to control the sample positioner, into the Kmax™ environment.

A cabling diagram for the data acquisition electronics is shown in Figure 5. Depicted in this diagram are several Nuclear Instrumentation Modules (NIM) as well as several CAMAC modules housed in a CAMAC "crate". X rays are detected with a Princeton Gamma-Tech (PGT) planar lithium-drifted silicon, or Si(Li), detector, which is not shown in the diagram. Each X ray produces a small charge pulse in the detector crystal, which is pre-amplified and shaped before being sent through coaxial cable to the main amplifier, an Ortec Model 572. The pulse from the main amplifier is finally directed to the Ortec Model AD114 ADC.

The time required to process pulses in the amplifier and the ADC is a concern for high count rate data acquisition. The amplifier requires 6 μs to fully process a pulse, and the ADC requires an additional 5 μs to digitize it. If two X rays enter the detector within the 6-μs resolving time of the amplifier, a summed-energy pulse is presented to the ADC and this produces pileup events in the spectrum, which increase the background. Excess background complicates fitting the data. Also, if a second pulse is presented to the ADC

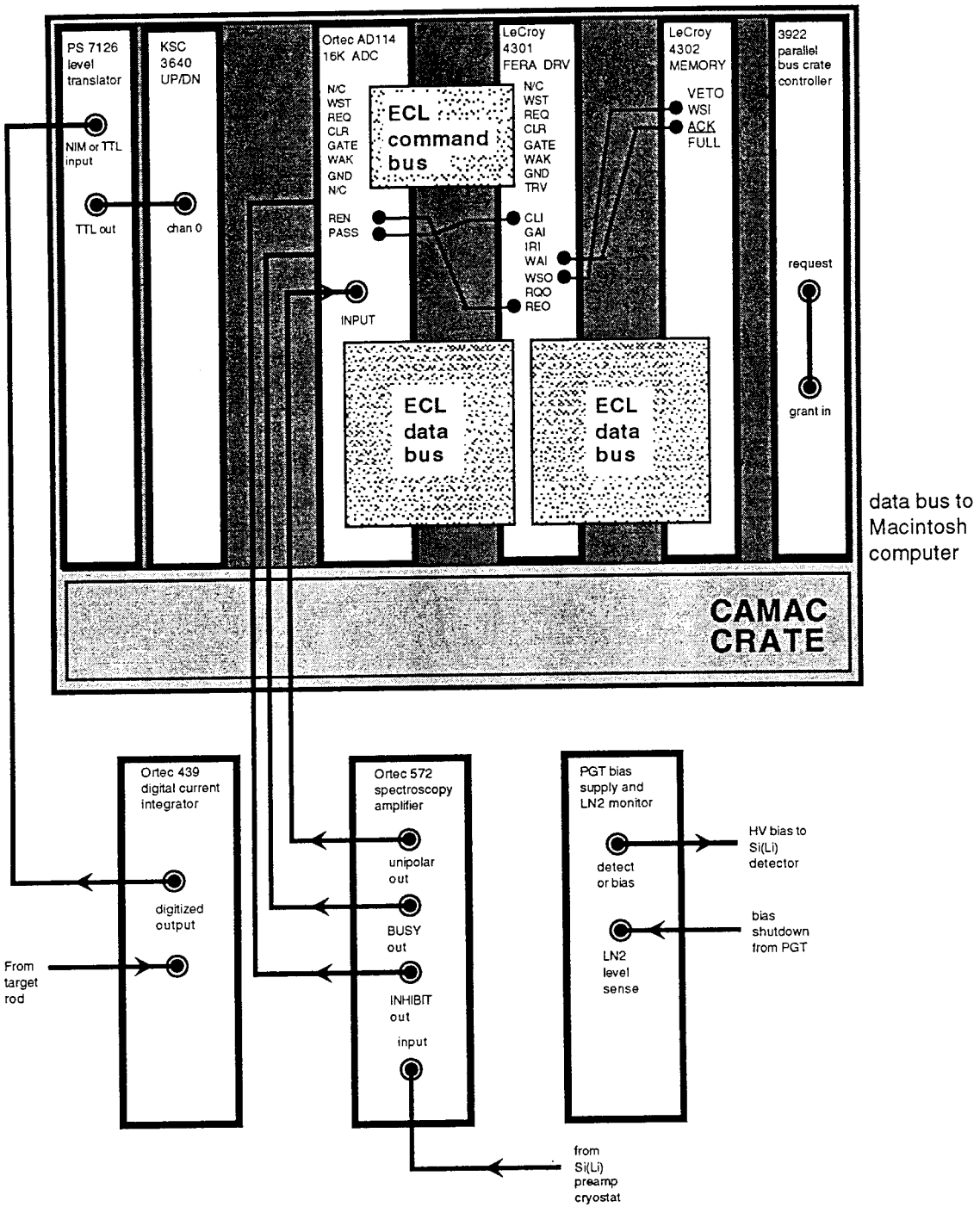


Figure 5 Data acquisition electronics and cabling for the NIM and CAMAC modules.

while it is digitizing a previous one, the later pulse cannot be processed and may actually detract from the quality of the conversion.

Both problems may be reduced significantly by making use of control signals produced by the electronic modules. The amplifier contains circuitry to detect pulse pileups, and it generates an INHIBIT signal when pileup occurs. The ADC produces a BUSY signal during its conversion period. By connecting the INHIBIT and BUSY signals between the Ortec 572 amplifier and the AD114 ADC, spectra that were acceptably free of pulse pileup or other distortions were obtained at event rates up to about 7.5 kHz, which is ten times the maximum event rate possible if these control signals were not used.

Due to the large number of processes monitored by a Macintosh CPU, the speed at which the Macintosh computer can respond to requests (interrupts) by the CAMAC crate is limited [Pi94]. This problem is not unique to Macintosh CPUs, but is also known to occur in most other CPU types (IBM PC and DEC LSI series). This was found to limit the maximum data-acquisition rate early in the project when the microprobe instrument source code only implemented non-buffered, single-event transfers into the Macintosh computer's random access memory.

By buffering the data transfer between the CAMAC crate and the CPU, one can increase the event rate by several orders of magnitude (typically from 300 Hz to more than 10 kHz). To provide such buffering, the AD114 ADC is connected to the 4301 Fast Encoding and Readout ADC (FERA) driver and the 4302 FERA memory through a high-speed Emitter-Coupled Logic (ECL) command bus and data bus. Instead of the AD114 requesting that each individual event be serviced by the Macintosh operating system directly, the AD114 transfers data on the ECL bus to the FERA memory under the supervision of the FERA driver. Between memory transfers to the Macintosh computer's random access memory, the FERA memory will store up to 16K events of data. When

the FERA memory is almost full it will request service from the Macintosh operating system. The Macintosh operating system will then service the request by doing a buffered memory transfer from the FERA memory directly into the its own memory, which is significantly faster than a series of single-event transfers from the AD114 ADC.

A Macintosh serial port driver XCMD was purchased from Sparrow Corporation and installed into the Kmax™ environment. This readily allowed the development of source code in Kmax™ to coordinate X-ray spectra acquisition with sample positioning controlled by the same software instrument.

The first functional data acquisition "instrument" was completed in December 1994. Its user interface window is shown in Figure 6. The first tests of this instrument involved measuring the beam spot size using Transmission Electron Microscope (TEM) grids. These measurements are described in Chapter III. Since the completion of the first instrument, much of the effort in the project has been devoted to testing and optimizing code. A flowchart of events occurring in the present data acquisition instrument is shown in Figure 7.

Documented source code and details of possible interest to future developers are given in Appendix I.

Spectra are saved by the Kmax™ program in a packed format. Filenames are constructed in the form: MMDDHHVV.kmx; where MM is the month the data was taken, DD the day, and HH and VV the horizontal and vertical scan coordinates. Although the packed data format saves a large amount of disk space and is useful for long-term storage, it is not suitable for input to the PIXE analysis programs. Steps required to prepare the data sets for further analysis are described in detail in Appendix II.

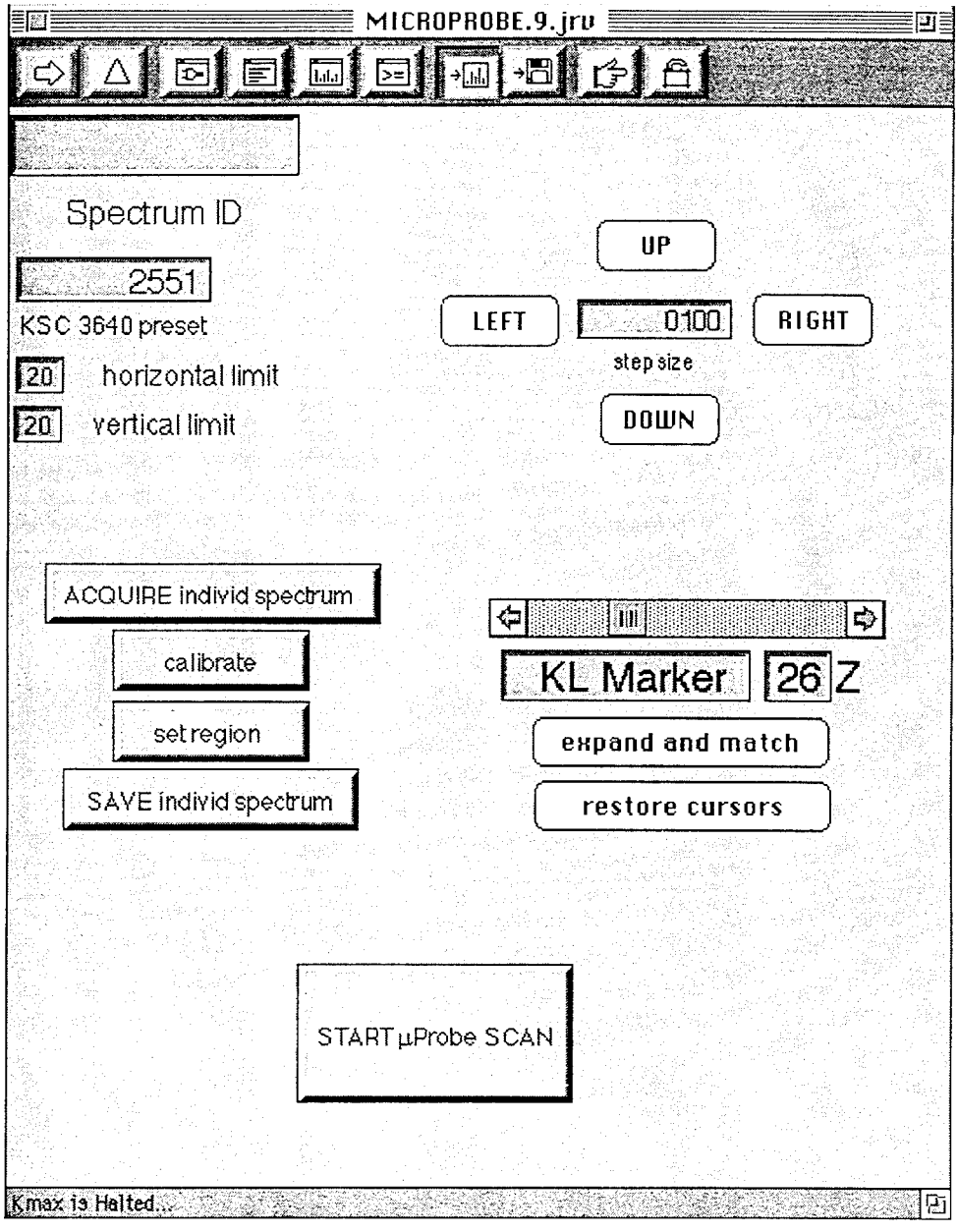


Figure 6. The Macintosh user interface window for data acquisition using the scanning ion microprobe.

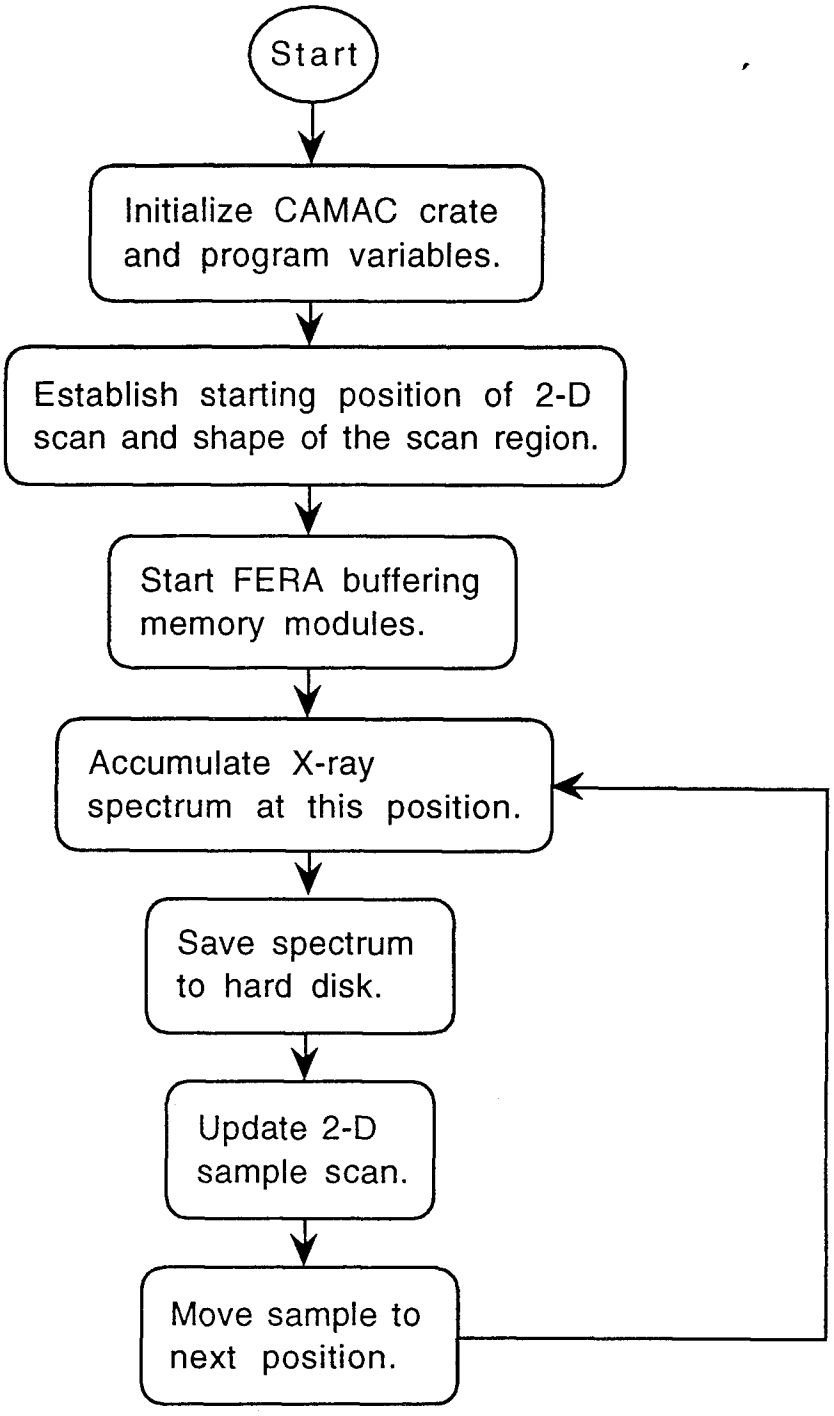


Figure 7. Outline of the steps used to take data with the microprobe instrument.

## II.C. Data Reduction

Several hundred X-ray spectra are generated in a typical two dimensional scan of a sample. Each individual spectrum is a complex entity containing the X-ray peaks of up to 20 elements superimposed on a background that may vary strongly with energy. Frequently the peaks corresponding to different elements overlap. It is clear that elemental concentrations can only be extracted with the help of a computer. This is done by fitting the spectra to a mathematical model. Once the elemental concentrations are extracted from the PIXE spectra, the concentrations must be correlated with information about the position on the sample to which they correspond, and they must be displayed in a format that facilitates interpretation.

At NATALY, a program known as GUPIX [Ma93] is used to extract elemental concentrations from the PIXE spectra. GUPIX can be run in an automatic mode which fits all of the spectra for a two dimensional scan with minimal user interaction. In this mode, GUPIX writes an output file that contains the concentration and position information. This file is then converted to a format that is universally recognized by the data visualization packages on the Macintosh Quadra 950 and VAX-VMS computers in the laboratory.

GUPIX fits the PIXE spectra using a mathematical model that considers a typical PIXE spectrum to be a composite of the X-ray peak data superimposed on a background which may vary strongly with energy. GUPIX does not fit the spectral background; rather, it convolutes the raw spectrum with a digital filter to remove the slowly varying background component. It is this convoluted spectrum that GUPIX actually fits using an internal data base of X-ray energies, intensities and attenuation coefficients.

The mathematical model in GUPIX accounts for the fact that even monoenergetic X rays incident on a detector will not generate a sharp spike in the measured spectrum because of statistical fluctuations in the amount of charge liberated in the detector by the

X ray and collected by the detector circuitry. To model the response of the particular X-ray detector that was used, GUPIX describes each spectral peak as the sum of a Gaussian function plus several additional functions such as exponential tails and square steps.

Only the PGT X-ray detector (serial number OS26-9118) was used to obtain the data presented in this report. To develop a parameterization for the line shape as a function of atomic number for this detector, PIXE spectra were measured for pure samples of magnesium, aluminum, silicon, calcium, vanadium, iron, and copper. It was found that the line shape could be adequately modeled as a Gaussian function centered on the X-ray energy, with a variable width determined by the fitting program, plus one exponential tail on the low energy side. Data concerning the detector line shape is provided to GUPIX in an ASCII text file.

The convoluted spectra are fit using a nonlinear least squares fitting procedure. This analytic approach to the fitting of the PIXE spectra has several advantages. The program can make small corrections to the energy calibration supplied by the user, correct the relative intensities of the X-ray lines in the database for differential absorption in the target material, and identify and correct for corrupted energy peaks in the spectrum. Corrupted peaks are either "pile up" peaks (from several X rays arriving in the detector at very nearly the same time) or "escape" peaks (from individual X rays that are not completely processed in the detector). If not recognized and corrected for, these types of corrupted energy peaks can confuse the analyst.

Although GUPIX has many powerful features, it has some limitations as well. GUPIX can make only minor corrections to the energy calibration that the user provides. If the energy calibration is not good to begin with, it is very unlikely that GUPIX will be able to fit the spectrum well. Additionally, the program apparently cannot achieve high quality fits for spectra with both low-atomic-number and high-atomic-number elements, possibly because it uses a fixed filter width for background removal even though the

peaks of high-energy X rays are actually wider than those of low-energy ones. This was not found to be a significant problem in fitting the microprobe data, but it did make a systematic contribution to the error in the fitting procedure nonetheless.

X rays entering the detector must always pass through two thin windows. Because of their effect on the spectrum, these windows are generally referred to as "filters". A polyamide window provides permanent vacuum isolation between the detector cryostat and the interior of the RBS chamber. A second window made of beryllium functions as a light shield for the detector crystal. X rays entering the detector are attenuated by both windows as well as by the actual detector crystal.

After many measurements with pure elements and standards of known composition, it was learned that the mathematical modeling of attenuation in the GUPIX program was not adequate to reproduce the known concentrations accurately. Perhaps this should not have been a surprise, because the GUPIX program allows one to insert a correction factor called the H-value. Experience with the detectors used in previous research at NATALY had not revealed so serious a problem as the one encountered with the PGT detector.

To properly analyze the data obtained with the PGT detector, it was necessary to obtain an empirical formula expressing the correction to the detector efficiency as a function of atomic number. To obtain this empirical relation, additional spectra of pure elemental samples were measured and fit with GUPIX. For pure elemental samples, GUPIX should report that the concentration is very close to one million parts-per-million (ppm). For spectra obtained using the PGT detector, GUPIX reported concentrations for pure elemental samples that were systematically less than one million ppm and which varied strongly but smoothly from one element to another. The external correction factor (H-value) was calculated for each of the pure elements, plotted as a function of atomic number (Z) and fit with a non-linear least squares program to obtain an empirical

relationship between H-value and atomic number. The resulting relationship may be expressed as

$$\log_{10}(H) = 5.2439 - 19.956 \log_{10}(Z) + 9.1617 [\log_{10}(Z)]^2. \quad \text{(equation 1)}$$

This relationship is illustrated graphically in Figure 8.

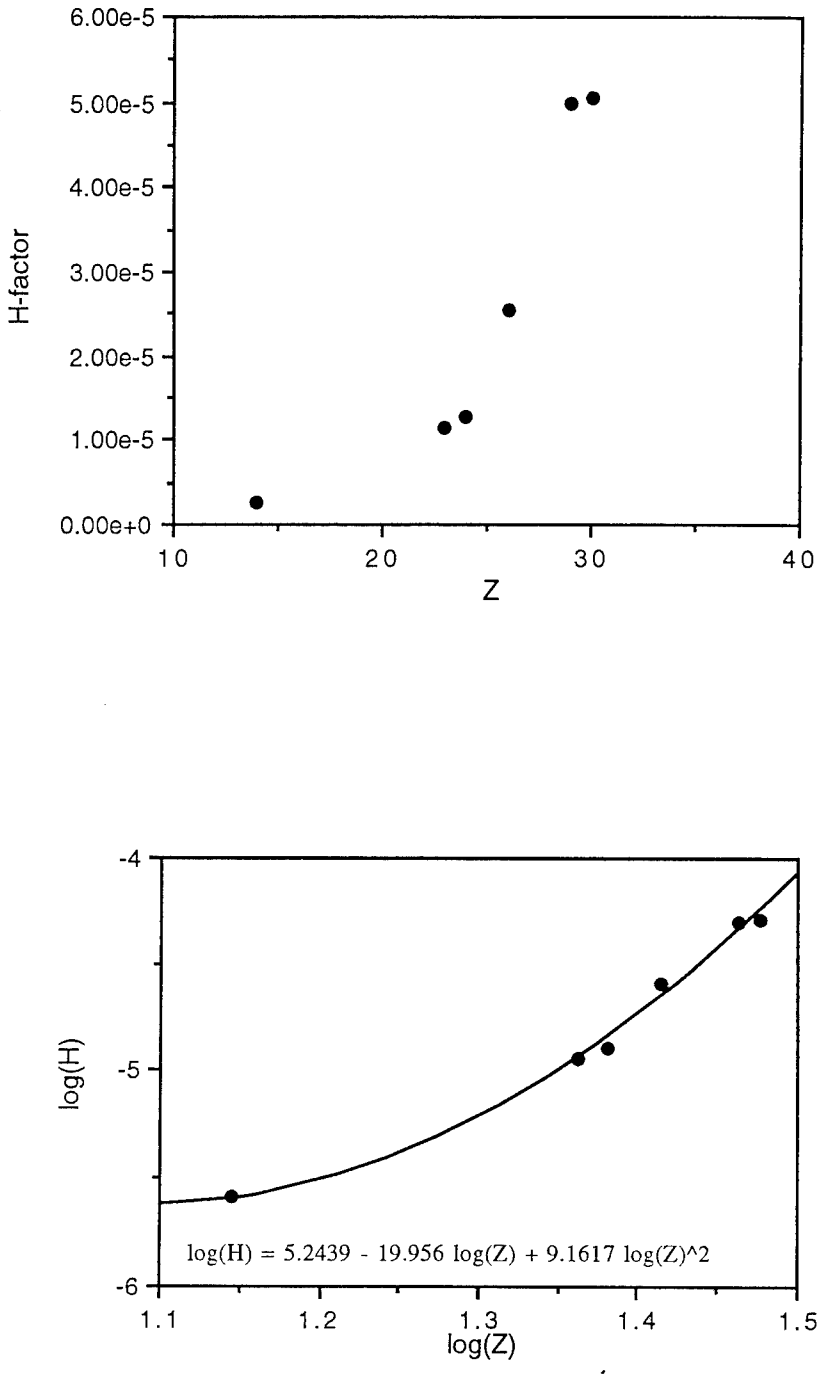


Figure 8. H-factor dependence on the atomic number, Z. The upper plot shows the variation in H with Z obtained using pure elements as samples. The lower plot shows an empirical second-order fit of $\log_{10}(H)$ to $\log_{10}(Z)$. This fit is used to compute H values for all elements and thereby extract concentration values from X-ray counts.

## III. Testing the Microprobe with a TEM Grid

Once the microprobe was constructed, it was necessary to measure its spatial resolution. This was accomplished by imaging a 200-mesh transmission electron microscope (TEM) grid with the microprobe. The 200-mesh grid has a lattice spacing of 127 μm and is made of wires that are about 60 μm thick. The resolution of the microprobe is determined by analyzing a two dimensional scan of the grid with the particle beam. All such measurements must be made under high vacuum in the RBS chamber because the atmosphere strongly scatters and attenuates the particle beam.

The copper TEM grid was mounted on an aluminum target holder in the RBS chamber. With this experimental setup, copper X rays are generated whenever a portion of the proton beam strikes the copper TEM grid wires, and aluminum X rays are produced whenever a portion of the beam strikes the aluminum backing between the wires. The two dimensional plot of copper X-ray counts versus position that was obtained experimentally is shown in Figure 9, which looks rather like a microscope view of the copper mesh. Because the number of detected copper X rays falls to zero between the grid wires, it may be deduced that the particle beam fits completely between the wires. From a detailed analysis of Figure 9, it was determined that the beam spot was between 30 and 40 μm in diameter.

During these tests, it was also learned that it is important to limit beam current to prevent damage to the target material. A highly focused particle beam with a current as low as 20 nanoamperes was observed to evaporate the thin copper wires of the TEM grid in less than a minute.

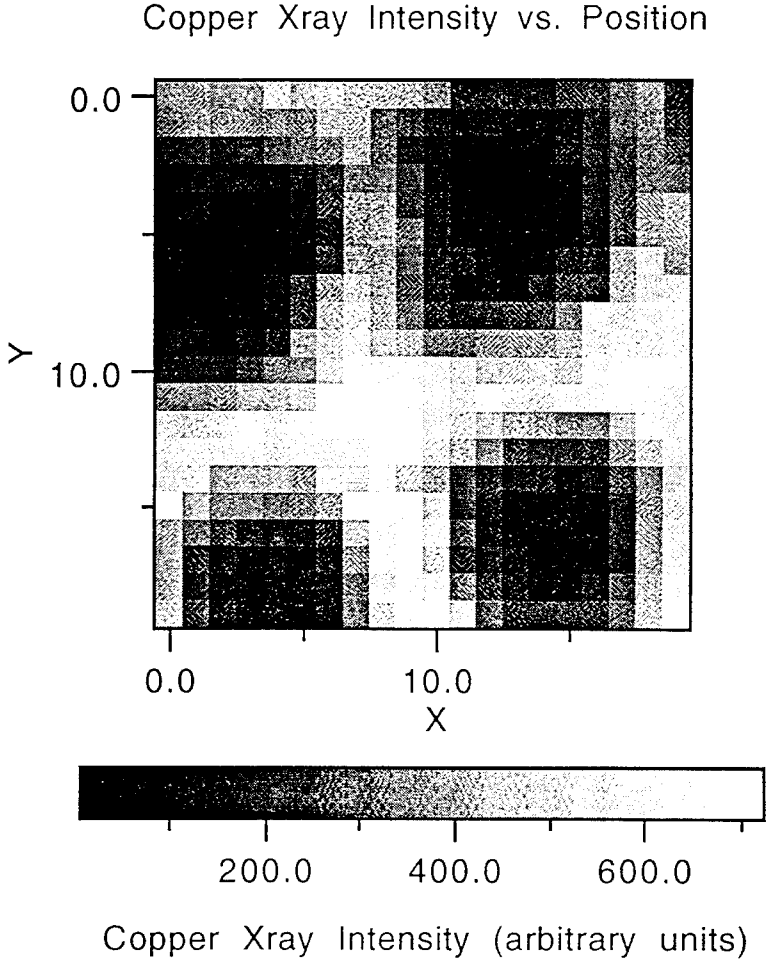


Figure 9. Copper X-ray counts versus position obtained from a microprobe scan of the TEM grid. The wires forming the grid are visible as white lines tilted with respect to the edges of the scan. The square dark spaces represent holes between the wires. The units for the X and Y axes are microprobe steps. Each microprobe step is 2.5 μm.

# IV. Measurements on the Allende Meteorite Inclusions

## IV.A. Background

Meteorites are remnants of the great cloud of gas and dust that formed the Sun and planets. Thousands of meteorites fall to the Earth's surface each year, but most strike the ocean or remote land areas and are not recovered. Many meteorites are thought to be fragments of asteroids, though some may be pieces from the heads of comets. Others seem uncorrelated with bodies in the solar system.

Meteorites provide interesting information about the composition of the Solar System. They may even provide information about the Earth's composition, because it is thought that they may be similar to material in the Earth's core and its mantle, which cannot be sampled directly.

This study focused on a particular class of meteorites called carbonaceous chondrites. Carbonaceous chondrites are stony meteorites with relatively large concentrations of carbon, which gives them their characteristic dark color. They also contain chondrules, or small, nearly spherical bodies consisting of various silicon oxides and metal-silicon oxides that are thought to predate the formation of the solar system approximately 4.6 billion years ago. Trace element analysis of the primitive material can provide information about the conditions that prevailed during the formation of the solar system [Mc85].

It is also useful to know the isotopic abundances of the elements present in meteorite samples [La93]. Measured isotopic abundances which deviate from standard solar system abundances may be due either to differences in the production mechanism of the isotopes or to some sensitivity of physical processes (such as transport or crystallization) to the isotopic composition. The radiation environment in interstellar space may also alter isotopic composition.

All elements with atomic mass numbers below 60 are formed by charged-particle reactions in stars, particularly those processes involving protons and alpha particles. The nucleogenesis of individual isotopes depends strongly on the temperature of the star: particle energies high enough to penetrate the Coulomb barrier of elements with atomic numbers above 26 (iron) are not, on average, available in the star at temperatures below $3.6 \times 10^9$ K.

Formation of isotopes with atomic mass larger than 60 depends strongly on neutron density inside the star. Heavy isotopes result from the slow-neutron capture process in second-generation stars and the rapid-capture process in supernovas. If the neutron density is on the order of $1 \times 10^8$ n/cm$^3$, then radioactive nuclei will usually beta-decay after capturing only one neutron, thus creating nuclei which lie along the valley of stability (s-process). If the neutron density reaches approximately $1 \times 10^{20}$ n/cm$^3$, the nuclei may capture several neutrons before beta-decaying, resulting in nuclei which lie far from the valley of stability (r-process). Hence knowledge of the relative pre-terrestrial isotopic abundances reveals characteristics (such as temperature and neutron density) of the early cosmos [Ro88].

A meteorite of particular interest is the Allende meteorite, which fell in Chihuahua, Mexico in February 1969. It is composed of an aluminum-silicate bulk material with a variety of chondrules [Gr85]. Some of the chondrules appear to have become mixed with the bulk material. This mixing possibly distorts the primitive record of elemental concentrations contained in the chondrules [Mc85]. Two dimensional maps of the composition in and around an individual chondrule can reveal the extent of mixing.

Elemental concentrations of fragments of the Allende meteorite have been measured by several researchers using chemical techniques [Ma 74], [Ka81]. Table I presents a summary of representative results from the literature, for comparison with the results of measurements to be discussed in the next section.

Table 1. Representative Literature Results for Allende Concentrations. All values given in parts per million (ppm) by weight.

| Element | Z | Powdered Sample[a] | Bulk Material[b] | Ca-Al Inclusions[c] |
|---|---|---|---|---|
| Sodium | 11 | 3290 | 3400 | 2900-3500 |
| Magnesium | 12 | 148000 | | 60600-99600 |
| Aluminum | 13 | 17600 | 17400 | 102000-233000 |
| Silicon | 14 | | 160000 | 83000-161000 |
| Potassium | 19 | 294 | 200 | 170-2200 |
| Calcium | 20 | 18800 | 1850 | 61400-171000 |
| Scandium | 21 | 11 | | |
| Titanium | 22 | | 900 | 1700-6400 |
| Vanadium | 23 | 99 | | |
| Chromium | 24 | 3630 | | |
| Manganese | 25 | 1450 | | |
| Iron | 26 | 237000 | 211200 | 10100-94900 |
| Cobalt | 27 | 662 | | |
| Nickel | 28 | 13300 | | |
| Zinc | 30 | 119 | | |

a) [Ka81] All uncertainties are given as < 5%.
b) [Ma74] Concentrations were derived after extracting chemical compounds from the sample. Uncertainties were not available.
c) [Ma74] Concentrations were derived after extracting chemical compounds from the sample.

IV.B. One Dimensional Scan

As a first example of the type of information that can be obtained by use of the scanning microprobe, the results of a one dimensional scan across the boundary of an inclusion will be presented. An inclusion in the Allende meteorite with a diameter of approximately 750 μm was selected which presented a well-defined circular cross section on the face of the meteorite. This inclusion was gray, with a smooth surface and dull luster, and it had a dark-colored ring surrounding the inclusion. The thickness of the ring was approximately 20 to 30 μm, and the ring was darker than either the exterior bulk material or material in the center of the inclusion.

Three scans with a 2.00-MeV proton beam were made across the boundary of the inclusion, covering a distance of 250 μm in 12.5 μm steps (reference name "4Meteor"). Based on experience gained in fitting other scans of the meteorite, two layers of 7.5 μm Kapton™ foil were used as an X-ray filter on the detector. This filter completely eliminated sodium and magnesium peaks from the spectrum and strongly attenuated aluminum peaks. The gain on the detector amplifier was adjusted so that the K-shell X rays of elements with atomic numbers up to that of arsenic (atomic number 33, $K_{\alpha}$ X-ray energy = 10.532 keV) could be observed. A sample spectrum and fit are shown in Figure 10. Concentrations averaged over the scan region are given in Table 2. The units are parts-per-million (ppm) by weight, and the number given in parentheses is standard deviation of the average.

It is instructive to compare the values presented in Table 2 with those in Table 1. The concentrations of titanium, chromium, manganese, and iron shown in Table 2 agree fairly well with the literature values of Table 1, giving confidence in the present results. However, concentrations of the lower-atomic-number elements silicon, potassium and calcium are higher in Table 2 than in Table 1, while that of the higher-atomic-number element nickel is lower in Table 2 than in Table 1. These differences from literature

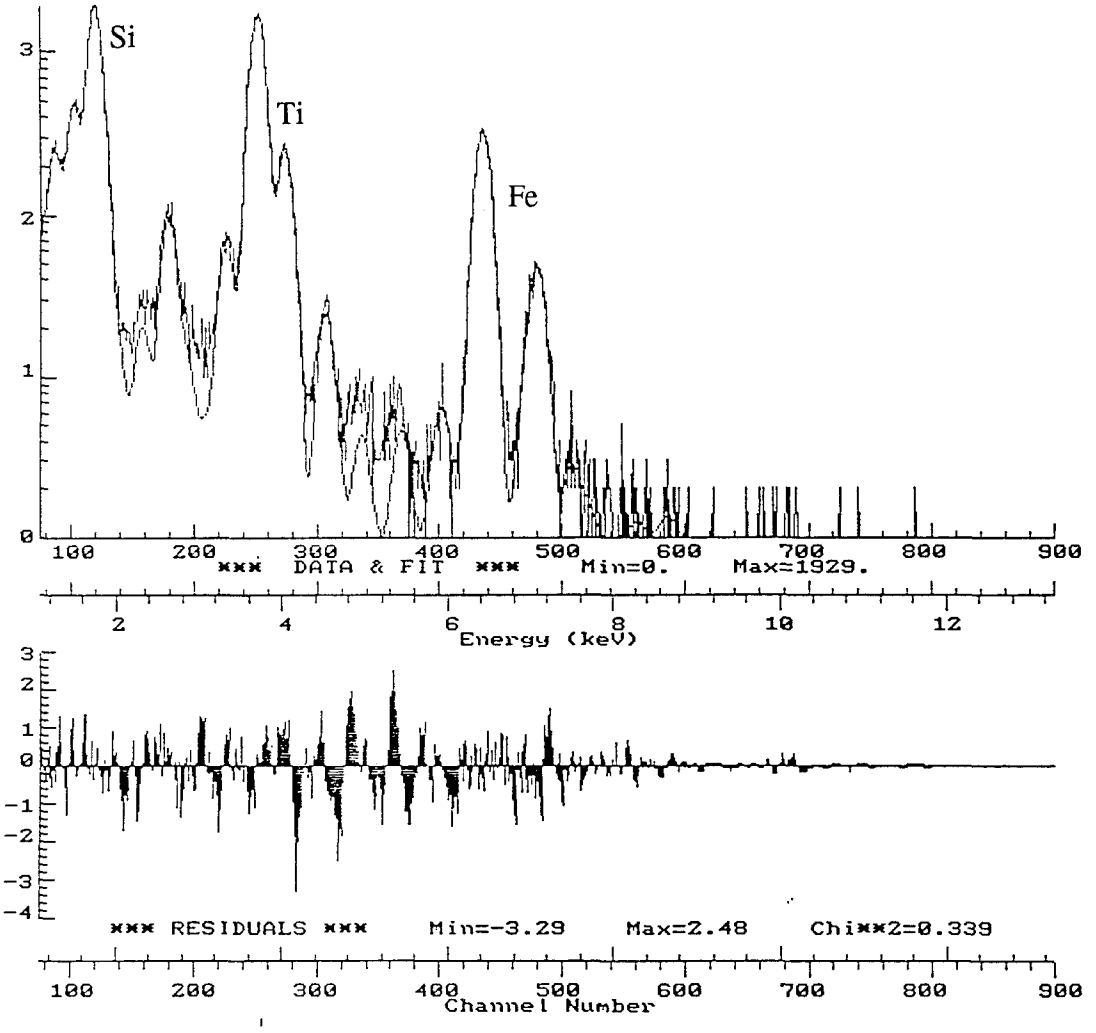


Figure 10. The upper plot shows a typical X-ray spectrum for a meteor sample (4Meteor) and the corresponding GUPIX fit . Deficiencies in the fit at low energies may be due to M-shell X rays or background processes that are not modeled in GUPIX. The lower plot illustrates residuals, which represent differences between the data and the fit expressed in units of standard deviations of the counts in the spectrum.

values may be genuine, because different inclusions do have different compositions, or they may indicate that an additional correction to the atomic-number dependence of the experimental sensitivity calibration is required.

Table 2. Average Concentrations in the one dimensional Scan "4Meteor". All values given in parts per million (ppm) by weight.

| Element | Z | Average Concentration | Limit of Detection |
|---|---|---|---|
| Silicon | 14 | 284500 (500) | 2000 |
| Sulfur | 16 | 80200 (200) | 200 |
| Chlorine | 17 | 16600 (100) | 700 |
| Potassium | 19 | 4880 (30) | 260 |
| Calcium | 20 | 24700 (50) | 260 |
| Titanium | 22 | 1020 (20) | 200 |
| Chromium | 24 | 3750 (20) | 140 |
| Manganese | 25 | 1440 (20) | 270 |
| Iron | 26 | 210300 (100) | 200 |
| Nickel | 28 | 7340 (30) | 120 |

To gain some understanding about variations of elemental concentration with position, concentrations were plotted versus scan location. Values for one scan are shown in Figure 11. The horizontal position labeled "0" lay outside the inclusion, in the bulk matrix, and the position labeled "20" was inside the inclusion, approximately half way out from the center. Because of difficulties orienting the sample and observing the position of the beam spot, the scan was not exactly radial.

Inspection of Figure 11 reveals several interesting features. First, the range of measurable concentrations spanned 3 orders of magnitude, showing the sensitivity of the PIXE technique. Second, the concentrations of sulfur (atomic number 16) and nickel

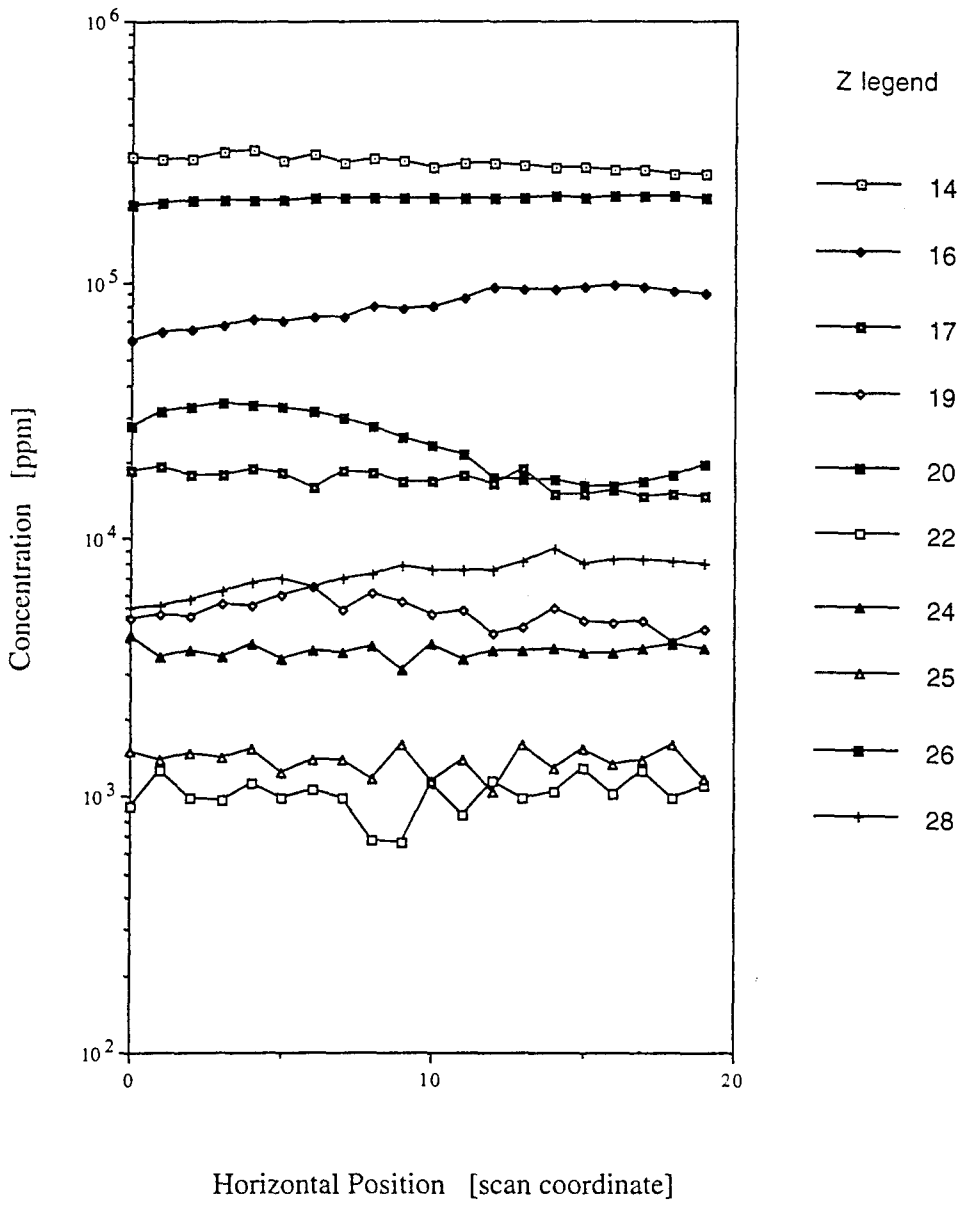


Figure 11. Concentration versus position for a one dimensional scan across the inclusion "4Meteor". The position labeled "0" is in the bulk material, while the position labeled "20" is in the interior of the inclusion, about halfway from the center to the edge.

(atomic number 28) are higher in the inclusion than in the surrounding bulk matrix, but the concentration of calcium (atomic number 20) is lower in the inclusion than in the matrix, clearly revealing the non-uniformity of the sample.

In order to more clearly demonstrate the relative changes in composition with position, the concentration of each element at each position was divided by the corresponding concentration at position "0" and the resulting ratios were plotted versus position. The results are shown in Figure 12. This Figure shows that the concentrations of sulfur and nickel are highly correlated across the entire scan range, and Table 2 gives an average ratio of sulfur to nickel concentration of approximately 10:1. Figure 12 also illustrates that the concentration of calcium apparently rises in the ring surrounding the inclusion, but falls sharply within the inclusion itself, and at a rate which is different from the rate at which the concentrations of sulfur and nickel rise.

It is significant that the beam spot size was approximately 30 μm, which corresponds to about three position steps. This means that although some rapid positional variations in the elemental concentrations may have been washed out because of the finite beam size, most of the large-scale variations shown in Figure 12 must be genuine. If data of this kind were obtained for a variety of similar inclusions, mechanisms of elemental migration between chondrules and the bulk matrix could be investigated.

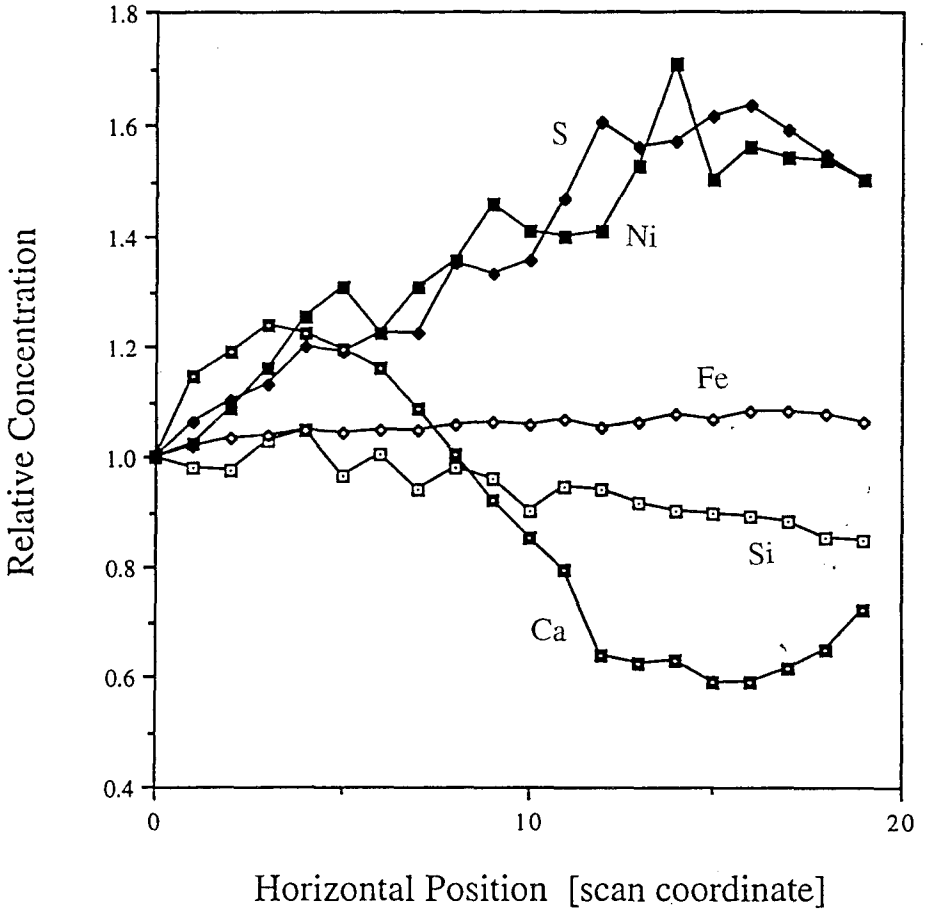


Figure 12. Relative concentrations versus position on the inclusion "4Meteor". For each element, the ratio of the concentration at each position to the concentration at position "0" is plotted. Position "0" is in the bulk material, while position "20" is in the interior of the inclusion, about halfway from the center to the edge.

## IV.C Two Dimensional Scans

Several two dimensional scans were performed to further test the microprobe and to demonstrate the variety of information that can be obtained with it. Many deficiencies in the original Kmax™ instrument became apparent during these scans and the instrument was modified accordingly.

A calcium-aluminum inclusion was selected on the face of the meteorite (reference name "1Meteor" 25Jan). This inclusion had an irregular shape and was approximately 750 μm × 1000 μm in size. The inclusion was composed of irregularly oriented crystals that were about the same size as the beam. There appeared to be some contamination from the bulk matrix in one location. A 15 × 15 grid was scanned using 2.30 MeV protons and a step size of 100 μm. The scan required 4 hours of beam time. The data was taken without additional filters on the X-ray detector.

Two dimensional scans contain a great deal of information, and it is difficult to know how to best present that information. Two methods were investigated. The first method was to create an X-ray "picture" of the scan region. Each such picture is a map of concentration versus position for a particular element in the sample. The images shown in Figures 13 and 14 were constructed from calcium and iron X rays using a commercial plotting package (Spyglass™ *Transform*). The location of the inclusion is readily apparent in these Figures: Figure 13 shows that the inclusion has a higher concentration of calcium than the surrounding material, whereas Figure 14 shows that it has a lower concentration of iron. Several image processing techniques provided with the Spyglass software were used on several maps of this kind to enhance edges or give an apparent increase in resolution, but such techniques lack numerical rigor or scientific basis, and the images they produced are not presented in this report.

A second technique investigated was to plot correlations between elemental concentrations at different points on the meteor. These plots were generated using

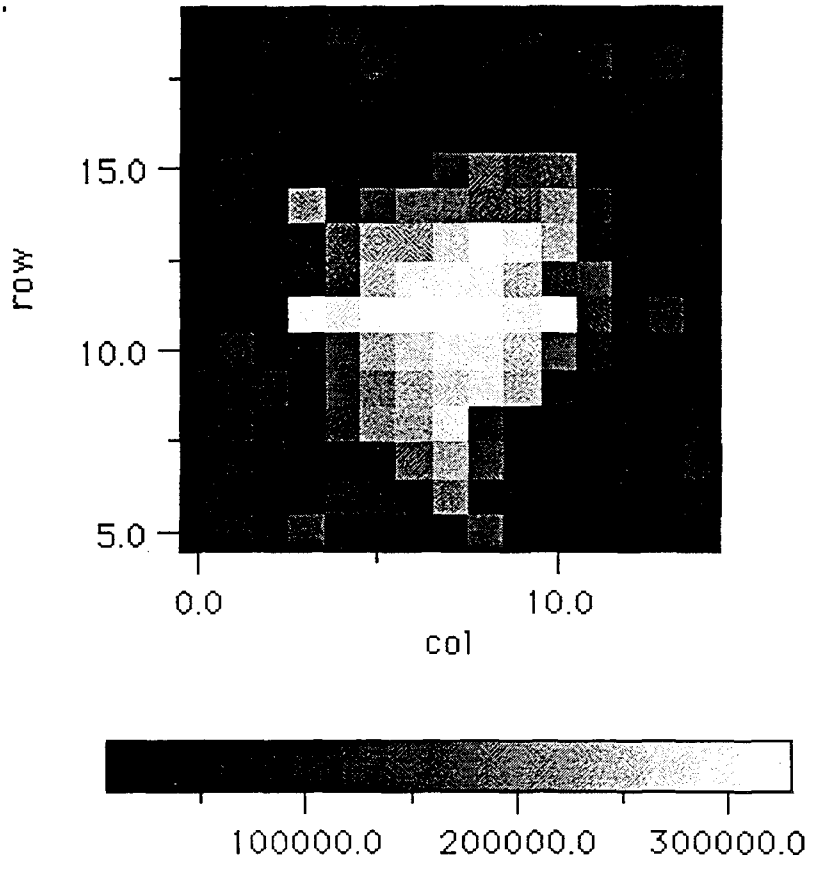


Figure 13. Calcium X-ray counts versus position for the inclusion "1Meteor". The units for the axes labeled ROW and COLUMN are goniometer steps. Each step represents 25μm.

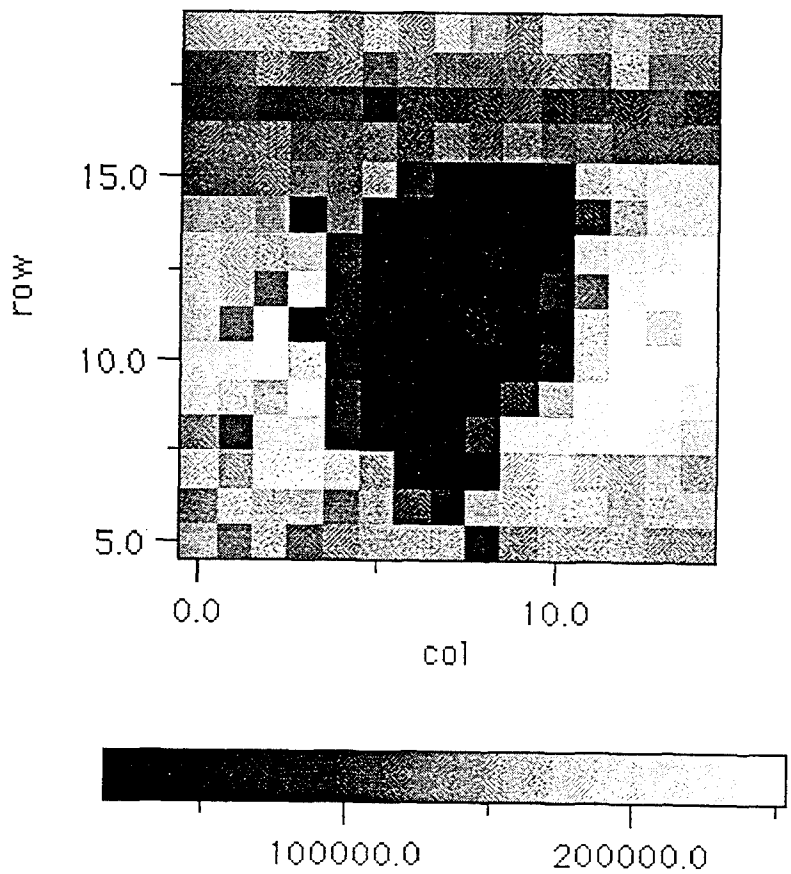


Figure 14. Iron X-ray counts versus position for the inclusion "1Meteor". The units for the axes labeled ROW and COLUMN are goniometer steps. Each step represents 25μm.

custom-written FORTRAN routines. Examples are shown in Figures 15 and 16. Figure 15 shows a plot of the concentration of nickel (atomic number 28) versus the concentration of sulfur (atomic number 16) at each scan location. Different plot symbols were chosen to indicate whether each point scanned was located inside the inclusion (points), outside the inclusion (squares), or on the boundary (crosses).

Figure 15 shows that the concentrations of sulfur and nickel are highly correlated at all points in the scan area, whether inside or outside the inclusion. The ratio of sulfur concentration to nickel concentration is apparently about 10:1, as it was for the inclusion "4Meteor" studied in the one dimensional scan.

Figure 16 shows the correlations between two additional pairs of elements in this inclusion. The upper plot shows that the concentration of silicon (atomic number 14) is about 10 times greater than that of sulfur (atomic number 16) outside the inclusion (squares), although the large scatter in the plotted squares indicates a wide variation in the ratio. Inside the inclusion (points) and on the boundary (crosses), the ratio of silicon to sulfur concentrations is much greater and less variable. The lower plot shows that the ratio of silicon to iron (atomic number 26) is approximately 2.5:1 outside the inclusion, but significantly greater, about 25:1 inside. Together, these plots reveal a silicon-rich inclusion, with small, but not randomly distributed, concentrations of sulfur and iron.

It is important to realize that the PIXE technique does not provide any direct information about the chemical bonding because chemical bond energies (in the range of eV) are very much smaller than the X-ray energies associated with transitions between the inner electronic orbits (keV). Further, the detectors used in PIXE measurements usually have energy resolutions on the order of 200 eV for a typical X-ray energy of 6 keV. Consequently, any effects of chemical bonding on X-ray transition energies are impossible to resolve. Clear correlations between elemental concentrations, as shown in Figures 15 and 16, are suggestive, however, and studies of the kind presented here have

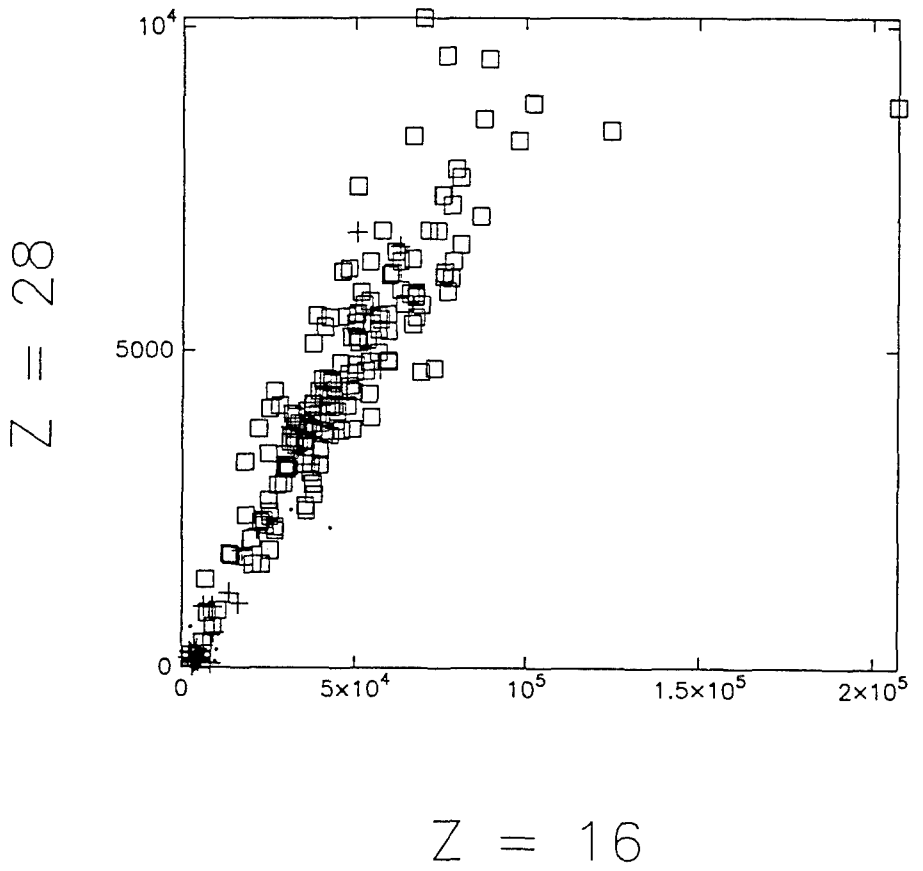


Figure 15. Concentration of nickel (Z = 28) plotted versus concentration of sulfur (Z = 16) for the inclusion "1Meteor". Concentration values are in parts per million (ppm) by weight. Square plotting symbols represent locations in the bulk matrix, points represent locations inside the inclusion, and crosses represent locations near the boundary.

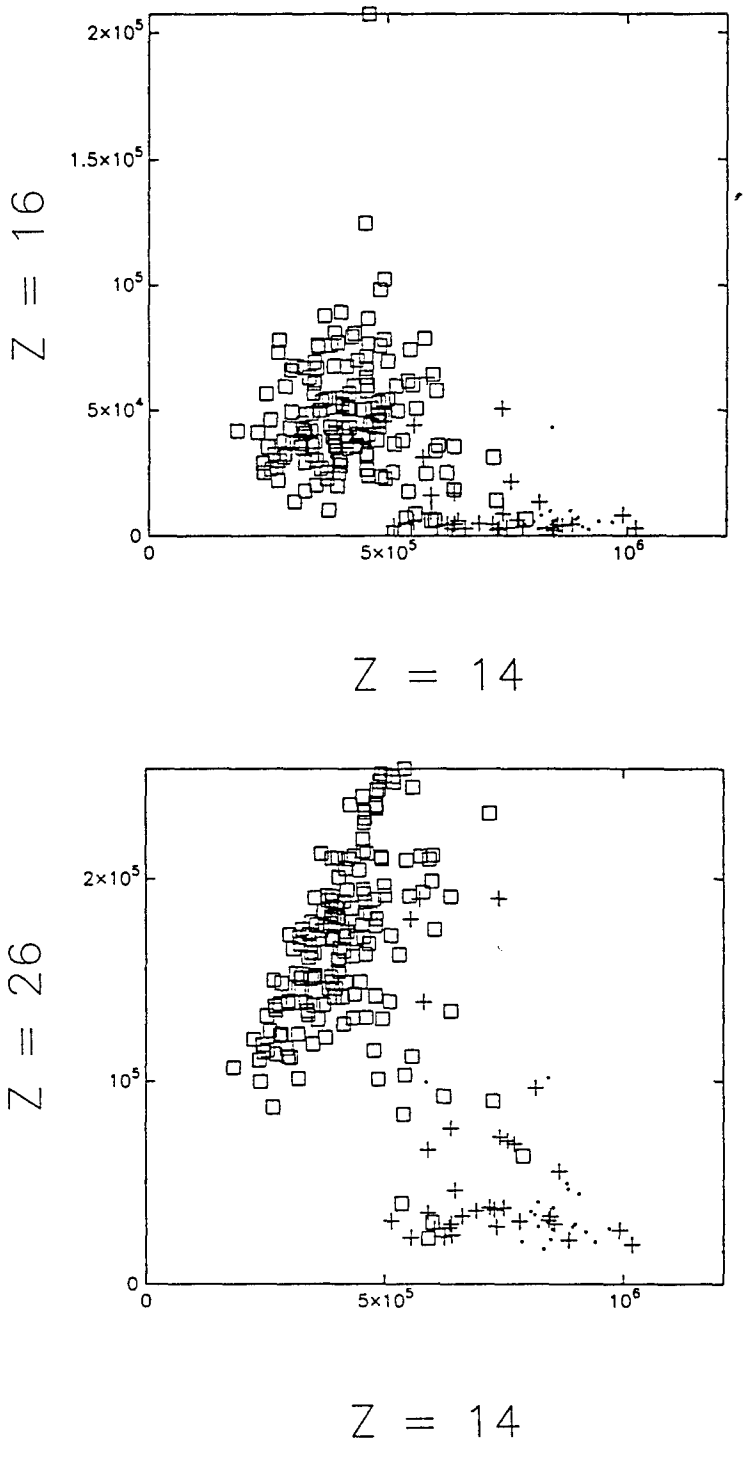


Figure 16. Top: Concentration of sulfur (Z = 16) plotted versus concentration of silicon (Z = 14) for the inclusion "1Meteor". Bottom: Concentration of iron (Z =26) versus concentration of silicon (Z = 14). Concentration values are in parts per million (ppm) by weight. Square plotting symbols represent locations in the bulk matrix, points represent locations inside the inclusion, and crosses represent locations near the boundary.

the potential to contribute greatly to the field of meteorite studies. The amount of information that can be generated in such scans is impressive: if there are N elements present in a sample, then there are N(N-1)/2 unique correlation plots. For N=21, which is typical for PIXE spectra, there are 210 different plots, each containing additional information.

As a final example, data is presented from the study of another inclusion, of a type known as a "Dark Clast" [He88] (reference name "3Meteor" 23Feb). Viewed with a microscope, the Dark Clast looked rather like a charcoal briquette, but it contained small crystals which strongly reflected light and it had deep cracks in several locations. A region containing the 1 mm × 1 mm nearly-round inclusion was scanned with 2.00-MeV protons in a 20 × 14 grid and a step size of 25 μm. The data required 5 hours of beam time to obtain, and it was taken without additional X-ray filters on the X-ray detector.

This scan clearly demonstrated that there are features in the meteorite that can be seen in X-ray images even though they are not apparent in a visual inspection. Although the microprobe scan had been set up to investigate the Dark Clast, it also revealed an unnoticed inclusion with large titanium concentrations on the very edge of the scan region. An X-ray image of the titanium distribution is shown in Figure 17.

Four correlation plots which display a variety of relationships are shown in Figures 18 and 19. The upper portion of Figure 18 demonstrates that the strong correlation between nickel and sulfur concentrations observed for "4Meteor" and for "1Meteor" is not universal: in "3Meteor", the concentrations of both nickel and sulfur are smaller than in the two previously studied inclusions, and the ratio of sulfur to nickel concentrations is generally higher though less well defined. The lower portion of Figure 18 shows that the concentrations of aluminum (atomic number 13) and manganese (atomic number 25) are completely uncorrelated. Conversely, the upper portion of Figure19 shows that the concentrations of titanium (atomic number 22) and iron (atomic

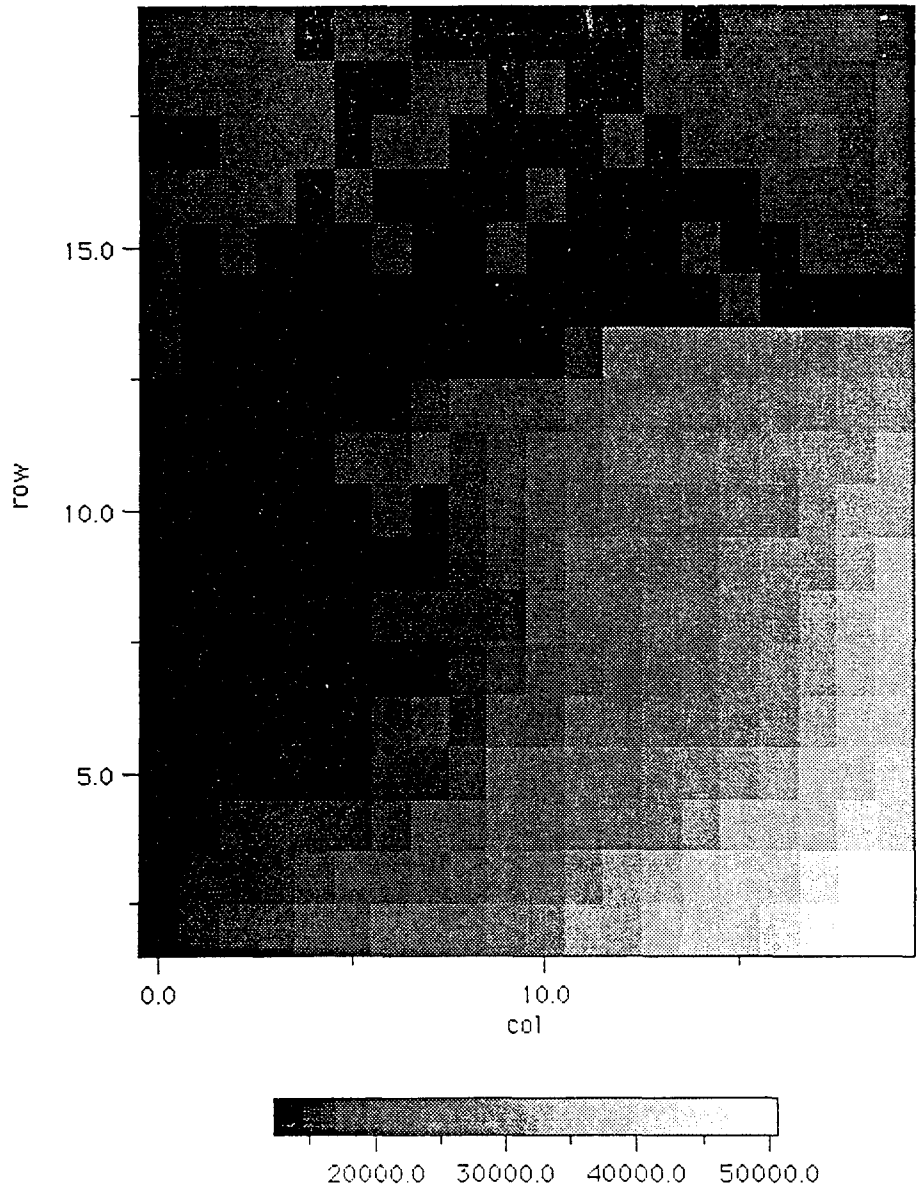


Figure 17. Titanium X-ray counts versus position for the inclusion "3Meteor". The units for the axes labeled ROW and COL are goniometer steps. Each step represents 25μm. The Dark Clast, which was the original subject of this scan, is in the upper left quadrant of the picture. A small, titanium-rich inclusion is in the lower right quadrant. It was not visible in a microscope examination of the sample.

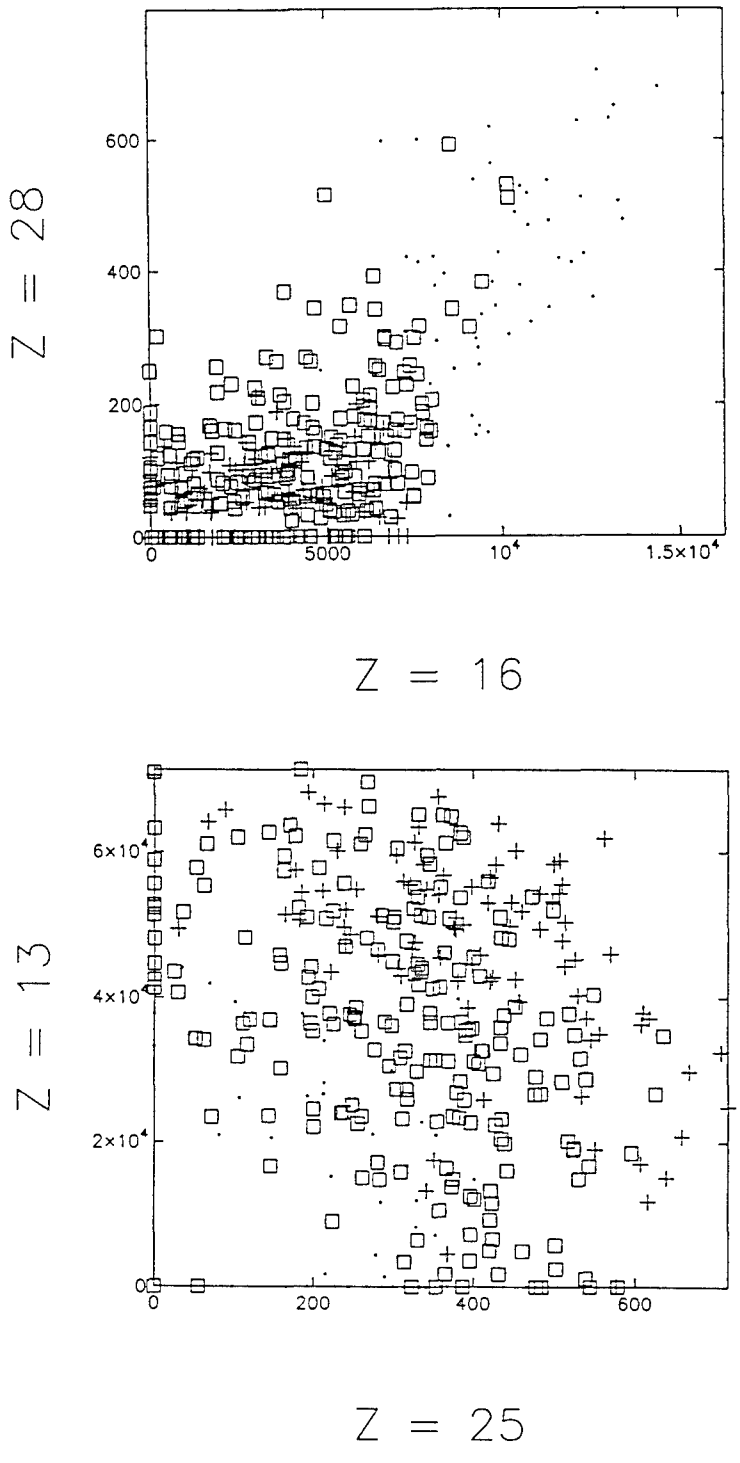


Figure 18 Top: Concentration of nickel (Z = 28) plotted versus the concentration of sulfur (Z = 16) for the inclusion "3Meteor". Bottom: Concentration of aluminum (Z = 13) versus concentration of manganese (Z = 25). Concentration values are in parts per million (ppm) by weight. Point plotting symbols represent locations in the titanium-rich portion and crosses represent locations in the Dark Clast.

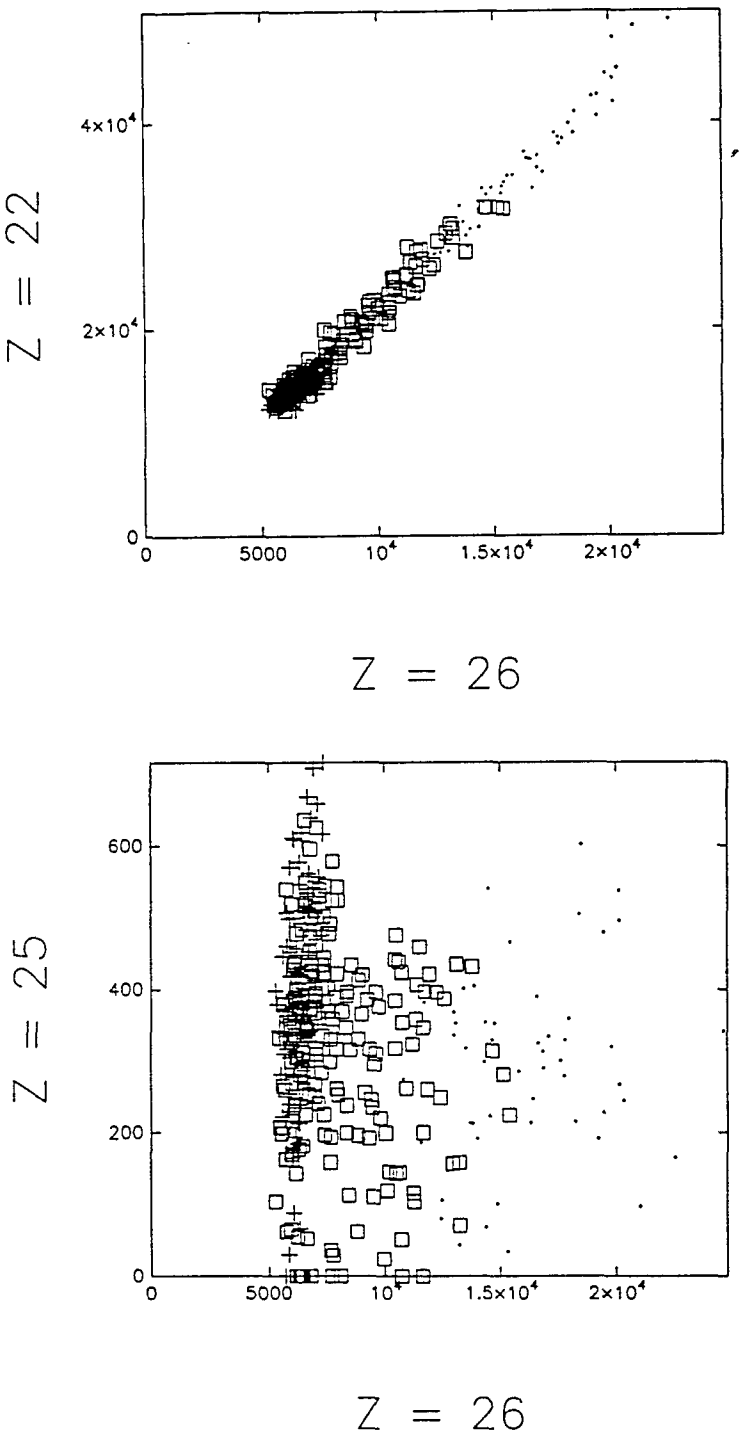


Figure 19. Top: Concentration of titanium (Z = 22) plotted versus the concentration of iron (Z = 26) for the inclusion "3Meteor". Bottom: Concentration of manganese (Z = 25) versus concentration of iron (Z = 26). Concentration values are in parts per million (ppm) by weight. Point plotting symbols represent locations in the titanium-rich portion and crosses represent locations in the Dark Clast.

number 26) are very strongly correlated, with a ratio of titanium to iron of about 2.2:1 in both the titanium-rich area (points) and the titanium-poor area (crosses) of the scan. The lower portion of Figure 19 illustrates two clear differences between the compositions of the titanium-rich and titanium poor areas: (1) the ratio of manganese (atomic number 25) to iron (atomic number 26) is much higher in the titanium-poor area, (2) and the iron concentration in that area is nearly constant, whereas it varies considerably in the titanium-rich area.

## IV.D. Preliminary Conclusions from Meteorite Studies

It is too early to draw extensive conclusions from the microprobe studies of the Allende meteorite. Nevertheless, some preliminary observations are appropriate.

It is apparent from the measurements made of the inclusions "1Meteor" and "3Meteor" that two dimensional X-ray maps are useful for imaging inclusions, even those that cannot be observed visually. In addition, plots correlating the concentration of one element with that of another illustrate a rich variety of relationships ranging from strict proportionality to an almost total lack of correlation. Different correlations are observed for different inclusions, as well as for the areas inside and outside an individual inclusion.

Similarly, one dimensional scans, such as those made of the inclusion "4Meteor", offer to provide useful information concerning the extent of elemental migration between chondrules and the surrounding matrix.

More detailed conclusions must await additional measurements and a more extensive data set.

## V. Summary

A scanning ion microprobe was developed which can be used to produce one and two dimensional maps of elemental concentrations across the surface of a sample. An essential part of the microprobe is a software "instrument" that coordinates sample positioning, data acquisition, and storage of the X-ray spectra obtained. That instrument was developed using the Kmax™ programming environment.

In order to evaluate the performance of the scanning ion microprobe, several data sets were acquired from samples with recognizable structure. First, a TEM grid with known grid spacing was used to measure the spatial resolution of the microprobe. The beam spot diameter on target was found to be approximately 30 μm. The microprobe was then used to scan several different types of inclusions in the Allende meteorite. In the process, several interesting elemental correlations were found, and many programming errors and design flaws were discovered and corrected.

Automated procedures were developed to extract concentrations from large PIXE data sets using the analysis program GUPIX. Although the GUPIX program was convenient to use, several deficiencies were discovered. Among the problems encountered were: (1) the present data base restricts analysis to X-ray lines from elements heavier than sodium (Z=11, $K_\alpha$ X-ray energy = 1.059 keV), (2) the program apparently cannot achieve high quality fits for spectra with both low-atomic-number and high-atomic-number elements, possibly because it uses a fixed filter width for background removal, and (3) the program did not treat special background situations such as the generation of spectral noise by secondary electron emission from the target.

Several methods to visualize the results were investigated, including a commercial software display package and several special-purpose, user-written routines. More

development is needed to perform detailed quantitative analysis, because of the large amount of information contained in the spectra.

The completed scanning ion microprobe has greatly extended the capabilities of the Naval Academy Tandem Accelerator Laboratory and provided a rather uncommon tool that will be applied to a variety of elemental analysis concerns in the future.

## VI. Acknowledgments

I would like to recognize and thank those individuals whose generous assistance and invaluable guidance made this project possible.

Mr. Charlie Holloway, the physics department machinist, helped us with the experimental setup. In many cases Mr. Holloway saved us much time and effort by pointing out flaws in our designs, and provided on the spot solutions to even our most persistent problems. I would also like to thank Mr. David Moore, the NATALY accelerator technician. Mr. Moore was instrumental in the completion of this project because he provided the expertise necessary to control the focusing and shaping of the particle beam. His many years of experience and intimate familiarity with our tandem accelerator system doubtlessly saved us weeks if not months in troubleshooting and maintenance of the accelerator.

I would also like to thank my advisors, Professors J. R. Vanhoy and F. D. Correll for their generous devotion of time and effort. Without their help I would surely still be wondering why in the world that nothing ever works. For their faith, patience, and tutelage they have my utmost gratitude.

Thank you all for your help.

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## VIII. BIOGRAPHY

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Abstracts:

"Development of a Scanning Ion Microprobe," B.T. Meehan, F.D. Correll, J.R. Vanhoy, Southeastern Section Meeting of the American Physical Society, Newport News, VA, 10 - 12 November 1994.

"Fragmentation of Isovector Strength in 142Ce," Sally F Hicks, C.M. Davoren, C.L. Lundstedt, J.R. Vanhoy, B.H. Benedict, B.M. Haas, B.T. Meehan, Bull Am. Phys. Soc 38, 1818 (1993). Asilomar Conference Center, Pacific Grove, CA, 21 - 23 October 1993.

Additional Training:

Kmax™ training session, Norfolk VA, 5 November 1994

Memberships:

Sigma Pi Sigma Physics Honor Society

Phi Kappa Phi Honor Society

Phi Alpha Theta History Honor Society

Sigma Xi

# Appendix I. Source code for the Kmax™ Instrument "Constant Charge.8.3"

## A: Source Code for the Kmax™ Data Acquisition Instrument

The current version of the microprobe software instrument is titled "MICROPROBE.9". Its source code is not presented here. Instead, the code for an earlier version is presented.

As originally written, the MICROPROBE.9 code was extensively documented to assist future researchers. Unfortunately, the software linker presently used to generate executable code can accept no more than 32K characters. MICROPROBE.9 eventually exceeded this limit, and most comments had to be removed to reduce its size.

The fully-commented code for an earlier version, titled ConstantCharge.8.3" is given below. When the new software linker is released in the summer of 1995, it will no longer have the 32K character restriction and comments may be reinserted.

```
; code for this instrument by Tim Meehan and Jeff Vanhoy
; version 8
; Friday the 13th   (January1995)

;=====CONTROLS=============================================
DECLARE C1 AS EVENT                                  ; display destructions
DECLARE C2 AS EVENT                                  ;the "DOWN" button
DECLARE C3 AS EVENT                                  ; the "UP" button
DECLARE C4 AS EVENT                                  ; the "LEFT" button
DECLARE C5 AS EVENT                                  ; the "RIGHT" button
DECLARE C6 AS EVENT                                  ; "one buffer" of info for calib and region choice
DECLARE C7 AS EVENT                                  ; change increment
DECLARE C8 AS EVENT                                  ; this is the  "spectrum ID" button
DECLARE C9 AS EVENT                                  ; "calibration" based on the one buffer of info
from detector
DECLARE C10 AS EVENT                                 ;  "start µprobe" button
DECLARE C11 AS EVENT                                 ; "set region" on the one buffer full.
DECLARE C12 AS EVENT                                 ; the "KL Marker" slider bar
DECLARE C13 AS EVENT                                 ; expand and match
DECLARE C14 AS EVENT                                 ; get cursors button
DECLARE C15 AS EVENT                                 ; set preset scalar
DECLARE C16 AS EVENT                                 ; set the number of horizontal steps per side
DECLARE C17 AS EVENT                                 ; set the number of vertical steps per side
DECLARE C25 AS EVENT                                 ; this is the KLM marker from the PIXIE program

;=====NON CONTROL SUBROUTINES=============================
DECLARE move_beam AS EVENT
DECLARE save_spectrum AS EVENT
DECLARE 2D_Data AS EVENT
DECLARE end_srq AS EVENT                          ; this is in a separate event for timing purposes


;=====SERIAL_OPEN Variables=================================
DECLARE port AS STRING
DECLARE baud AS INTEGER
DECLARE StopBits AS STRING
DECLARE buffhan AS INTEGER                         ; SERIAL_OPEN returns the input buffer pointer into
this
DECLARE status AS INTEGER                           ; SERIAL_OPEN will return the error code
DECLARE parity AS INTEGER
DECLARE inrefnum AS INTEGER                       ; SERIAL_OPEN returns inrefnum into parity
DECLARE DataBits AS INTEGER
DECLARE outrefnum AS INTEGER                      ; SERIAL_OPEN returns outrefnum into DataBits
DECLARE statusstr AS STRING                        ; SERIAL_OPEN will return the error message into this

;=====Variable Declarations================================
DECLARE text AS STRING
DECLARE text2 AS STRING
DECLARE in_string AS STRING
DECLARE z_axis AS BOOLEAN                          ; true if current axis is the z axis
DECLARE step_size AS STRING
DECLARE data AS INTEGER                             ; intermediate calculations
DECLARE increment AS INTEGER                      ;integer value of the step size ... not currently used.
DECLARE horizontal_limit AS INTEGER                                ; this is ONE MINUS the number of
beam spots.
DECLARE vertical_limit AS INTEGER                                 ; this is ONE MINUS the number of
beam spots.
DECLARE pointer AS INTEGER                          ; this variable contains the data pointer from the
FERAMEM

DECLARE h_steps AS INTEGER                          ; this is the loop counter for the horizontal
direction
DECLARE v_steps AS INTEGER                          ; this is the loop counter for the vertical
direction
DECLARE dir_right AS BOOLEAN                      ; this variable will implement the serpent trace on the
target
DECLARE running AS BOOLEAN                         ; running true if start milliprobe is set, otherwise
false.
DECLARE x_pos AS INTEGER
DECLARE y_pos AS INTEGER
DECLARE x_last AS INTEGER
DECLARE y_last AS INTEGER                           ; these are used in the Beam Position histogram
DECLARE fera_count AS INTEGER                     ; this is a variable used in a crude decision in SRQ
DECLARE spectrum_id AS STRING                     ; this is a variable used in the save_spectrum routine
DECLARE preset AS INTEGER                           ; this is the preset for the KSC 3640
DECLARE srq1 AS BOOLEAN
DECLARE srq2 AS BOOLEAN
DECLARE first_region_set AS BOOLEAN        ; this variable tests to see if the first region was set.
DECLARE second_region_set AS BOOLEAN     ; this variable tests to se if the second region was set.
;=====Variables used in Jeff's routines=========================
; variables used in the KLM marker routine
DECLARE z AS INTEGER
DECLARE ifind AS INTEGER
DECLARE nXrays AS INTEGER
DECLARE engy AS REAL
DECLARE loop AS INTEGER
DECLARE ichan AS INTEGER
; variables used in the calibration routine
DECLARE e1 AS REAL
DECLARE e2 AS REAL
DECLARE slope AS REAL
DECLARE intercept AS REAL
```

```
DECLARE leftcursor AS INTEGER
DECLARE rightcursor AS INTEGER
DECLARE temp1 AS REAL
DECLARE temp2 AS REAL
; variables used in the set region routine
DECLARE label AS STRING
;====GO==================================================
; initialize and inhibit the crate.
; set or initialize global variables
; This portion of the program allows initalizes the goniometer
; and checks to see if the SMC is responding to the Mac's serial commands.
;=========================================================
ON GO DO SEQUENCE
    SET horizontal_limit TO 19                                          ; this is ONE MINUS the
number of steps.
    SET vertical_limit TO 19                                           ; this is ONE MINUS the
number of steps.
    SET first_region_set TO FALSE
    SET second_region_set TO FALSE
    OPEN "Beam Position"
    OPEN "2D data"
    OPEN "KLM"
    OPEN "ap0klx.hist"
    OPEN "L Xray.hist"
    CLEAR "Beam Position"
    CLEAR "Report"
    BRING "param-1" TO FRONT
    SET XCALIBRATION TO 0 0                        ; remove stored calibration

    SET spectrum_id TO ""                             ; initialize the front panel indicators.
    SET label TO "region_name"
    SET TEXT TO "Spectrum ID//"
    CONCAT_STR TEXT spectrum_id
    SET_CONTROL 8 TEXT

    SET step_size TO "0100"
    SET TEXT TO "step size//"
    CONCAT_STR TEXT step_size
    SET_CONTROL 7 TEXT

    SET TEXT TO "/horizontal limit//"
    SET data TO horizontal_limit
    SET data TO vertical_limit
    ADD 1 TO data
    SET text TO data
    CONCAT_STR TEXT text
    SET_CONTROL 16 TEXT

    SET TEXT TO "/vertical limit//"
    SET data TO vertical_limit
    ADD 1 TO data
    SET text TO data
    CONCAT_STR TEXT text
    SET_CONTROL 17 TEXT

    SET preset TO 2551                                 ; the default for the preset value
    SET text TO preset
    SET TEXT TO "KSC 3640 preset//"
    CONCAT_STR TEXT text
    SET_CONTROL 15 TEXT

    RECORD "initializing CAMAC crate . . ."
    CAMIO <30,0,17> ( 7 )                              ;KSC 3922 CC: clear, initialize and inhibit
    CAMIO <30,13,17> ( 4194308 )                  ;KSC 3922 CC: LAM mask 4, 23

    CAMIO <4,0,24>                                      ; KSC 3640 COUNTER: Disable LAM Requests

    CAMIO <21,0,16> ( 7424 )                       ; AD 114 ADC: sequential non COINC, ECL, no gate
    CAMIO <21,0,26>                                   ; AD 114 ADC: Enable ADC.

    CAMIO <23,1,17> ( 1 )                             ; FERA MEM: enable CAMAC mode.
    CAMIO <23,0,17> ( 0 )                             ; FERA MEM: Write memory Address Ptr.
    CAMIO <23,0,26>                                    ; FERA MEM: Enable LAM.
    CAMIO <23,1,17> ( 3 )                             ; FERA MEM: enable ECL mode.
    FLUSH

    CAMIO <30,13,17> ( 4194312 )                  ; KSC 3922 CC: LAM mask 4, 23
    RECORDLN "done."

    SET h_steps TO 1                                    ; initialize the beam position histo & vars
    SET v_steps TO 1
    SET x_pos TO 0
    SET y_pos TO vertical_limit
    SET x_last TO 0
    SET y_last TO vertical_limit
    SET PORT TO "Beam Position"
    SET DATA TO 10
    SET CHANNEL x_pos y_pos
    SET fera_count TO 0

    SET port TO "MODEM"                               ; serial port defaults
    SET baud TO 4800
```

```
    SET parity TO 0
    SET DataBits TO 8
    SET StopBits TO "1"
    RECORD "Opening Input/Output drivers . . ."
    SERIAL_OPEN   baud  StopBits  parity  DataBits  buffhan  status  statusstr
    WAIT 100 MSECS                                          ;allow time for the SMC to give info
                                                                  ;Get Input and Output reference
numbers
    SET inrefnum TO parity
    SET outrefnum TO DataBits

    SET text TO "1QA"                                  ; this tests to see if the SMC is on
    CONCAT_STR text 13
    SERIAL_WRITE  text  outrefnum  status  statusstr  ; outrefnum from SERIAL_OPEN
    WAIT 500 MSECS                                          ;allow time for the SMC to give info

    SERIAL_READ   in_string  inrefnum  status  statusstr
    WAIT 500 MSECS                                          ;allow time for the SMC to give info
    SERIAL_READ   in_string  inrefnum  status  statusstr
    WAIT 500 MSECS                                          ;allow time for the SMC to give info
    IF (status <> 0) THEN
        PLAY_SOUND "SMC"
    END IF

    SET text TO "1WP0010"                          ; sets z axis as the starting axis
    CONCAT_STR text 13
    SERIAL_WRITE   text  outrefnum  status  statusstr
    WAIT 500 MSECS                                           ;allow time for the SMC to give info
    SET z_axis TO TRUE
    SET dir_right TO TRUE                                ; I want to scan right first

    SET running TO FALSE                                 ; you will want to set up the instrument before
taking data, right?
    RECORDLN "Done."
END SEQUENCE

;=====Set spectrum_id=====================================
ON C8 DO SEQUENCE
    SET TEXT TO spectrum_id
    PROMPT WITH "Spectrum Identification\n\n\t -- Enter Month, Day (mmdd)"
    IF OK THEN
        SET spectrum_id TO TEXT
        SET TEXT TO "Spectrum ID//"
        CONCAT_STR TEXT spectrum_id
        SET_CONTROL 8 TEXT
    END IF
END SEQUENCE

;=====MANUAL CONTROL SECTION=============================
; After the SMC is initialized, it is then necessary for the beam position to be
; initialized on the target.  This section allows the use of the arrow
;buttons on the instrument to position the beam.  Less error trapping
; than the SMC Manual Driver instrument was implemented to save time.

;=====Start µprobe Button================================
; this button is pressed after you are done positioning
ON C10 DO SEQUENCE
    CLEAR "param-1"
    SET running TO TRUE
    CAMIO <30,0,17> ( 4 )                              ; KSC 3922 CC: set source inhibit.
    CAMIO <23,1,17> ( 1 )                              ; LRS 4302 FERA Mem: enable CAMAC mode.
    CAMIO <23,0,17> ( 0 )                              ; LRS 4302 FERA Mem: reset data pointer to zero.
    CAMIO <23,1,17> ( 3 )                              ;LRS 4302 FERA Mem: enable ECL mode
    CAMIO <4,0,10>                                       ; KSC 3640 COUNTER: clear LAM in counter 0
    CAMIO <4,0,16> (preset)                            ; KSC 3640 COUNTER: write preset to counter 0
    CAMIO <4,0,26>                                       ; KSC 3640 COUNTER: Enable LAM Requests
    CAMIO <30,0,17> ( 0 )                              ;KSC 3922 CC: clear source inh
END SEQUENCE

;=====The Down Button====================================
ON C2 DO SEQUENCE
    IF ( z_axis = FALSE )  THEN                          ; ensures that the current axis is the z axis
        SET z_axis TO TRUE                               ; this makes a note that the current axis is
the z axis.
        SET text TO "1WP0010"
        CONCAT_STR text 13
        SERIAL_WRITE   text  outrefnum  status  statusstr
        WAIT 500 MSECS                                          ;allow time for the SMC to give info
        SERIAL_READ   in_string  inrefnum  status  statusstr
        WAIT 500 MSECS                                   ;allow time for the SMC to give info
        RECORDLN "selected z"
    END IF
    SET text TO "1MP"
    CONCAT_STR text step_size
    RECORD "command="
    RECORD text
    RECORDLN " (DOWN)"
    CONCAT_STR text 13
    SERIAL_WRITE   text  outrefnum  status  statusstr ; this writes "1MP/-in_string(+return)" to the
SMC
    WAIT 750 MSECS                                       ; allows time for the SMC to respond
    SERIAL_READ   in_string  inrefnum  status  statusstr
```

```
    WAIT 750 MSECS                                          ; allows time for the SMC to respond
END SEQUENCE

;=====The Up Button=========================================
ON C3 DO SEQUENCE
    IF ( z_axis = FALSE )  THEN                        ;select z axis if z_axis is not the current axis.
        SET z_axis TO TRUE                               ; this makes a note that the current axis is
the z axis.
        SET text TO "1WP0010"
        CONCAT_STR text 13
        SERIAL_WRITE   text  outrefnum  status  statusstr
        WAIT 500 MSECS                                          ;allow time for the SMC to give info
        SERIAL_READ   in_string  inrefnum  status  statusstr
        WAIT 500 MSECS                                     ;allow time for the SMC to give info
        RECORDLN "selected z"
    END IF
    SET text TO "1MR-"
    CONCAT_STR text step_size
    RECORD "command="
    RECORD text
    RECORDLN " (UP)"
    CONCAT_STR text 13
    SERIAL_WRITE   text  outrefnum  status  statusstr ; this writes "1MR-//in_string//(return)" to the
SMC
    WAIT 750 MSECS                                          ;allows time for the SMC to respond.
    SERIAL_READ   in_string  inrefnum  status  statusstr
    WAIT 750 MSECS                                          ;allows time for the SMC to respond.
END SEQUENCE

;=====The Left Button========================================
ON C4 DO SEQUENCE
    IF ( z_axis = TRUE )  THEN                         ; this will change the current axis to the x
axis if it is not selected.
        SET z_axis TO FALSE                              ; this makes a note of the current axis
        SET text TO "1WP0001"
        CONCAT_STR text 13
        SERIAL_WRITE   text  outrefnum  status  statusstr
        WAIT 500 MSECS                                          ;allow time for the SMC to give info
        SERIAL_READ   in_string  inrefnum  status  statusstr
        WAIT 500 MSECS                                     ;allow time for the SMC to give info
        RECORDLN "selected x"
    END IF
    SET text TO "1MR-"
    CONCAT_STR text step_size
    RECORD "command="
    RECORD text
    RECORDLN " (LEFT)"
    CONCAT_STR text 13
    SERIAL_WRITE   text  outrefnum  status  statusstr ; this writes "1MR//in_string//(return)" to the
SMC
    WAIT 750 MSECS                                          ;allows time for the SMC to respond.
    SERIAL_READ   in_string  inrefnum  status  statusstr
    WAIT 750 MSECS                                          ;allows time for the SMC to respond.
END SEQUENCE

;=====The Right Button=======================================
ON C5 DO SEQUENCE
    IF ( z_axis = TRUE)  THEN                          ; this will make sure that the current axis is
the x axis.
        SET z_axis TO FALSE                              ; this makes a note that the current axis is the
x axis.
        SET text TO "1WP0001"
        CONCAT_STR text 13
        SERIAL_WRITE   text  outrefnum  status  statusstr
        WAIT 500 MSECS                                          ;allow time for the SMC to give info
        SERIAL_READ   in_string  inrefnum  status  statusstr
        WAIT 500 MSECS                                     ;allow time for the SMC to give info
        RECORDLN "selected x"
    END IF
    SET text TO "1MR"
    CONCAT_STR text step_size
    RECORD "command="
    RECORD text
    RECORDLN " (RIGHT)"
    CONCAT_STR text 13
    SERIAL_WRITE   text  outrefnum  status  statusstr ; this writes "1MR//in_string//(return)" to the
SMC
    WAIT 750 MSECS                                          ;allows time for the SMC to respond.
    SERIAL_READ   in_string  inrefnum  status  statusstr
    WAIT 750 MSECS                                          ;allows time for the SMC to respond.
END SEQUENCE

;=====The Increment Button===================================
; the increment event is used to define the manual step increment
; for the motor while the user is manually positioning the beam
; on the target.  The increment is used only in the L/R/U/D button
; group at the top righthand part of the instrument palette.

ON C7 DO SEQUENCE
    SET TEXT TO step_size
    PROMPT WITH "Input the new increment for the motor.                    Must be <= 5000!"
    GET step_size TO TEXT
```

```
    SET increment TO step_size                          ; converts "inc_string" to an integer format
    IF (increment >= 5000) THEN
        REQUEST C7
    END IF
    LEN_STR   step_size  data                          ; returns the length of the string
    IF ( data = 1 ) THEN
        SET text TO "000"
        CONCAT_STR   text  step_size
        SET step_size TO text
    END IF
    IF ( data = 2 ) THEN
        SET text TO "00"
        CONCAT_STR   text  step_size
        SET step_size TO text
    END IF
    IF ( data = 3 ) THEN
        SET text TO "0"
        CONCAT_STR   text  step_size
        SET step_size TO text
    END IF
    SET TEXT TO "step size//"
    CONCAT_STR TEXT step_size
    SET_CONTROL 7 TEXT
END SEQUENCE

;=====SRQ subroutine=====================================
; the SRQ subroutine will deal with the hardware in the crate
;========================================================
ON SRQ DO SEQUENCE
    SET PAUSE TIMER TO 8 SECS
    CAMIO <30,0,17> ( 4 )                              ; KSC 3922 CC: set source inhibit.
    CAMIO <30,12,1>                                    ; KSC 3922 CC: Read LAM pattern.
    AND DATA WITH 4194312                             ; throw out any except slot 4 and 23
    SET data TO DATA

    CAMIO <22,0,9>                                     ; LRS 4301 FERA DRVR: COMMAND ECL -clear-.

    CAMIO <23,1,17> ( 1 )                             ; LRS 4302 FERA Mem: enable CAMAC mode.
    CAMIO <23,0,24>                                    ; LRS 4302 FERA Mem: Disable LAM.
    CAMIO <23,0,10>                                    ; LRS 4302 FERA Mem: Test and clear LAM.
    CAMIO <23,0,2>                                     ; LRS 4302 FERA Mem: Read memory, decrement
address.
    SET pointer TO DATA
    IF ( pointer > 16384 ) THEN
        SET pointer TO 16384
    END IF
    SET PORT TO "param-1"
    CAMIO <23,0,17> ( 0 )                             ; LRS 4302 FERA Mem: reset data pointer to zero.
    BUFIO < 23 , 0 , 0 > ( pointer , 1)
    UPDATE "param-1"

    IF (data = 8) THEN                                  ;if the SRQ was from the counter
        SET srq1 TO TRUE                               ;so that you can move to the next data point
    END IF

    IF (running = TRUE) THEN
        REQUEST 2D_Data
        IF (srq1 = TRUE) THEN
            REQUEST save_spectrum                              ;call before move_beam; uses vars
x_pos and y_pos .
            REQUEST move_beam
            CAMIO <4,0,10>                                        ; KSC 3640: clear LAM in counter 0
            CAMIO <4,0,16> ( preset )                         ; KSC 3640 COUNTER: Write counter 0
with preset
            CAMIO <4,0,26>                                    ; KSC 3640 COUNTER: Enable LAM
Requests
            SET srq1 TO FALSE
        END IF
    END IF

    REQUEST end_srq
END SEQUENCE

ON end_srq DO SEQUENCE
    CAMIO <23,0,17> ( 0 )                             ; LRS 4302 FERA Mem: reset data pointer to zero.
    CAMIO <23,0,26>                                    ; LRS 4302 FERA Mem: Enable LAM.
    CAMIO <23,1,17> ( 3 )                             ; LRS 4302 FERA Mem: enable ECL mode.
    CAMIO <30,0,17> ( 0 )                                 ;KSC 3922 CC: remove inhibit
    SET PAUSE TIMER TO 0 SECS
    ADD 8000000 TO DATA
                                                                ;    RECORD "SRQ LOOP TIME: "
                                                                ;    RECORD DATA
                                                                ;    RECORDLN " µsecs"
END SEQUENCE

;======Move_beam subroutine=============================
; this subroutine will be the loop that moves the beam
; also presently, code has been added to the subroutine that
; supports the beam position histogram.

ON move_beam DO SEQUENCE
    IF [illegible] THEN
```

```
        ADD 1 TO h_steps
        IF ( dir_right = TRUE) THEN
            CALL C5                                          ; this is a call to the move left
subroutine ...

            SET PORT TO "Beam Position"
            SET x_last TO x_pos
            SET y_last TO y_pos
            ADD 1 TO x_pos
            SET DATA TO 10
            SET CHANNEL x_pos y_pos
            SET DATA TO 1
            SET CHANNEL x_last y_last

        ELSE                                                   ; else the direction was left ...
            CALL C4                                          ; this is a call to the move right
subroutine

            SET PORT TO "Beam Position"
            SET x_last TO x_pos
            SET y_last TO y_pos
            SUBTRACT 1 FROM x_pos
            SET DATA TO 10
            SET CHANNEL x_pos y_pos
            SET DATA TO 1
            SET CHANNEL x_last y_last

        END IF
    ELSE
        SET h_steps TO 1

        IF ( dir_right = TRUE ) THEN
            SET dir_right TO FALSE
        ELSE
            SET dir_right TO TRUE
        END IF

        IF ( v_steps <= vertical_limit ) THEN
            ADD 1 TO v_steps
            CALL C2                                          ; this is a call to the move down
subroutine ...

            SET z_axis TO FALSE                              ; this reselects the x_axis as the
current axis.
            SET text TO "1WP0001"
            CONCAT_STR text 13
            RECORDLN "selecting the 'x' axis"
            SERIAL_WRITE   text  outrefnum  status  statusstr
            WAIT 500 MSECS                                          ;allow time for the SMC to give
info
            SERIAL_READ   in_string  inrefnum  status  statusstr
            WAIT 500 MSECS                                        ;allow time for the SMC to give info

            SET PORT TO "Beam Position"
            SET y_last TO y_pos
            SET x_last TO x_pos
            SUBTRACT 1 FROM y_pos
            SET DATA TO 10
            SET CHANNEL x_pos y_pos
            SET DATA TO 1
            SET CHANNEL x_last y_last

        ELSE
            CAMIO <30,0,17> ( 4 )                    ;KSC 3922 CC:Write Control/Status register.
            CALL HALT
        END IF
    END IF
    CLEAR "param-1"
END SEQUENCE
;=====Save_spectrum=======================================
ON save_spectrum DO SEQUENCE
    SET text TO spectrum_id
    SET text2 TO x_pos
    IF ( x_pos < 10 ) THEN
        CONCAT_STR text "0"
    END IF
    CONCAT_STR text text2
    SET text2 TO y_pos
    IF ( y_pos < 10 ) THEN
        CONCAT_STR text "0"
    END IF
    CONCAT_STR text text2
    CONCAT_STR text ".kmx"
    SET text2 TO ":datafolder:"
    CONCAT_STR text2 text
    SAVE "param-1" AS text
END SEQUENCE
;=====OD Data Event=======================================
ON OD_Data DO SEQUENCE
    SET POINT TO "param-1"
    GET REGION "data"
    REGION AREA "param-1" DATA
```

```
    SET PORT TO "2D data"
    SET CHANNEL x_pos y_pos
    RECORDLN DATA

    IF (second_region_set = TRUE) THEN
        SET PORT TO "param-1"
        GET REGION "data2"
        REGION_AREA "param-1" DATA
        SET PORT TO "2D data#2"
        SET CHANNEL x_pos y_pos
    END IF
END SEQUENCE

;=====KLM Marker (Jeff)=====================================
; this routine was taken from the PIXIE program without modification
; and integrated into the program.

ON C12 DO SEQUENCE
    CLEAR "KLM"
    SET z TO DATA
    SET_CONTROL 13 z
    SET PORT TO "ap0klx.hist"
    GET CHANNEL z
    SET nXrays TO DATA
    SET loop TO 1
    SET  ifind TO z
    SUBTRACT 1 FROM ifind                          ;histograms start from chan 0, not 1
    REPEAT
        SET PORT TO "ap0klx.hist"
        ADD 100 TO ifind
        GET CHANNEL ifind
        SET engy TO DATA
        DIVIDE engy BY 1000
        SUBTRACT intercept FROM engy
        DIVIDE engy BY slope
        SET ichan TO engy
        ADD 1000 TO ifind
        GET CHANNEL ifind
        SET PORT TO "KLM"
        SET CHANNEL ichan
        SUBTRACT 1000 FROM ifind
        ADD 1 TO loop
    UNTIL (loop > nXrays)
    SET PORT TO "param-1"
    OVERLAY "KLM"
    SET ifind TO z                                         ;beginning of the L alpha Xray section
    SUBTRACT 1 FROM ifind                          ;histograms start from 0, not 1
    SET PORT TO "L Xray.hist"
    GET CHANNEL ifind                                  ;get the first L Xray
    SET engy TO DATA
    DIVIDE engy BY 1000                            •   ; converts eV to keV
    SUBTRACT intercept FROM engy
    DIVIDE engy BY slope
    SET ichan TO engy
    ADD 300 TO ifind
    GET CHANNEL ifind                                   ; puts the relative amplitude in DATA
    SET PORT TO "KLM"
    SET CHANNEL ichan                                  ;sets the histogram bin ichan to DATA
    UPDATE ALL
    SET ifind TO z                                         ;beginning of the L beta Xray section
    SUBTRACT 1 FROM ifind                          ;histograms start from 0, not 1
    ADD 100 TO ifind                                    ;gets the second value
    SET PORT TO "L Xray.hist"
    GET CHANNEL ifind                                  ;get the first L Xray
    SET engy TO DATA
    DIVIDE engy BY 1000                                ; converts eV to keV
    SUBTRACT intercept FROM engy
    DIVIDE engy BY slope
    SET ichan TO engy
    ADD 300 TO ifind
    GET CHANNEL ifind                                   ; puts the relative amplitude in DATA
    SET PORT TO "KLM"
    SET CHANNEL ichan                                  ;sets the histogram bin ichan to DATA
    SET ifind TO z                                         ;beginning of the L gamma Xray section
    SUBTRACT 1 FROM ifind                          ;histograms start from 0, not 1
    ADD 200 TO ifind                                    ;gets the third value
    SET PORT TO "L Xray.hist"
    GET CHANNEL ifind                                  ;get the first L Xray
    SET engy TO DATA
    DIVIDE engy BY 1000                                ; converts eV to keV
    SUBTRACT intercept FROM engy
    DIVIDE engy BY slope
    SET ichan TO engy
    ADD 300 TO ifind
    GET CHANNEL ifind                                   ; puts the relative amplitude in DATA
    SET PORT TO "KLM"
    SET CHANNEL ichan                                  ;sets the histogram bin ichan to DATA
END SEQUENCE

;=====Energy Calibration (Jeff)=============================
ON C9 DO SEQUENCE                                      ; Single-Pk Energy Calibration
    SET PORT TO "param-1"
```

```
    ALERT WITH "Are Cursors Set To Peak Centroids Already?"
    IF OK THEN
        PROMPT WITH "ENTER Energy of Lower Peak"
        IF OK THEN
            SET  e1 TO TEXT
            PROMPT WITH "ENTER Energy of Upper Peak"
            IF OK THEN
                SET e2 TO TEXT
            END IF
            GET_CURSORS leftcursor rightcursor
            SET temp1 TO rightcursor
            SUBTRACT leftcursor FROM temp1
            SET temp2 TO e2
            SUBTRACT e1 FROM temp2
            DIVIDE temp2 BY temp1
            SET slope TO temp2
            SET temp1 TO e2
            MULTIPLY temp2 BY rightcursor
            SUBTRACT temp2 FROM temp1
            SET intercept TO temp1
            RECORD "RESULTS OF LINEAR ENERGY CALIBRATION\r"
            RECORD slope
            RECORD "\r"
            RECORD intercept
            RECORD "\r"
                                                        ;SET CALIBRATION TO slope intercept
            SET XCALIBRATION TO slope intercept
            SET PORT TO "KLM"
            SET XCALIBRATION TO slope intercept
            SET PORT TO "newinstrument"
        END IF
    END IF
END SEQUENCE

;=====Make Region (Jeff)==================================
; the program is only set up to handle 2 regions AT MOST.
; This routine shouldn't let you set more than two regions, but
; the program should work if only one region is set.

ON C11 DO SEQUENCE                          ; allows the user to set or modify a region
    IF (first_region_set = FALSE) THEN
        SET PORT TO "param-1"
        ALERT WITH "Are Cursors Set To Region Limits Already?"
        IF OK THEN
            GET_CURSORS leftcursor rightcursor
            SET TEXT TO "data"
            NEW REGION "data" AT leftcursor rightcursor
        END IF
    ELSE
        IF (second_region_set = FALSE) THEN
            SET PORT TO "param-1"
            ALERT WITH "Are Cursors Set To Region Limits Already?"
            IF OK THEN
                GET_CURSORS leftcursor rightcursor
                SET TEXT TO "data2"
                NEW REGION "data2" AT leftcursor rightcursor
            END IF
        ELSE                                        ; this condition satisfied if region
#1 AND #2 are set
            ALERT WITH "This program is only set up to handle 2 regions!"
        END IF
    END IF
END SEQUENCE

;=====Expand and Match Button=============================
ON C13 DO SEQUENCE
    SET PORT TO "param-1"
    GET_CURSORS leftcursor rightcursor
    SET PORT TO "KLM"
    SET CURSORS TO leftcursor rightcursor
    EXPAND "KLM"
    EXPAND "param-1"
END SEQUENCE

;=====Get Cursors Button==================================
ON C14 DO SEQUENCE
    SET PORT TO "param-1"
    OVERLAY ""
    UPDATE "param-1"
END SEQUENCE

;=====Set Preset Scalar===================================
ON C15 DO SEQUENCE
    SET TEXT TO preset
    PROMPT WITH "input new value for PSC?640 preset"
    SET text TO TEXT
    IF OK THEN
        SET preset TO text
        SET TEXT TO "PSC 3640 preset/ "
        CONCAT_STR TEXT text
        SET_CONTROL 15 TEXT
    END IF
```

```
END SEQUENCE

;=====Steps per side control=================================
ON C16 DO SEQUENCE
    SET data TO horizontal_limit
    ADD 1 TO data
    SET TEXT TO data
    PROMPT WITH "input value for top horizontal histogram index"
    IF OK THEN
        SET horizontal_limit TO TEXT
        SUBTRACT 1 FROM horizontal_limit
        SET data TO TEXT
        SET text TO data
        SET TEXT TO "/horizontal limit//"
        CONCAT_STR TEXT text
        SET_CONTROL 16 TEXT
    END IF

    SET h_steps TO 1                                    ; initialize the beam position histo & vars
    SET v_steps TO 1
    SET x_pos TO 0
    SET y_pos TO vertical_limit
    SET x_last TO 0
    SET y_last TO vertical_limit
    SET PORT TO "Beam Position"
    SET DATA TO 10
    SET CHANNEL x_pos y_pos
    SET fera_count TO 0
END SEQUENCE

ON C17 DO SEQUENCE
    SET data TO vertical_limit
    ADD 1 TO data
    SET TEXT TO data
    PROMPT WITH "input value for top vertical histogram index"
    IF OK THEN
        SET vertical_limit TO TEXT
        SUBTRACT 1 FROM vertical_limit
        SET data TO TEXT
        SET text TO data
        SET TEXT TO "/vertical limit//"
        CONCAT_STR TEXT text
        SET_CONTROL 17 TEXT
    END IF
END SEQUENCE

;=====get data button=========================================

END SEQUENCE
```

B: Sample Manipulator commands

The computer-controlled sample manipulator accepts serial commands. The microprobe instrument program issues commands to the sample manipulator using the Macintosh computer's serial modem line. The sample manipulator is microprocessor controlled, and although it can be programmed to perform simple tasks, it is much easier to issue commands directly from the Macintosh computer.

Illustrated below is the syntax of several commands that are useful for controlling the sample manipulator. All of the commands are ASCII strings, and all strings must start with the numeral "1."

1QA query status.

When this command is issued, the sample manipulator will return the current internal settings. The serial buffer must be cleared before the command is issued, otherwise the buffer will overflow. If the buffer was cleared, it must be read twice in order to retrieve all of the information.

1 MR (sign) (steps) move relative.

The command syntax is the ASCII string "1MR" followed by the optional sign character ("-" if a backward move is desired; nothing if a forward move is desired) and 4 characters that represent the number of steps that the controller is to move. Each step is 2.5 μm. The (steps) portion of the command MUST be 4 characters long; for instance, to move 10 steps the (steps) string would be 0010. Once the command is received and understood, the controller will return the string "OK" to the Macintosh serial buffer.

1WP(axis) select axis.

This command instructs the controller to select one of its 5 axes to be "active". All subsequent move commands will move the active axis. The axis string is an ASCII string of gray-coded numbers that refer to axes 0 through 4. The present microprobe instrument uses only two of the three axes that are connected to the controller. The microprobe program refers to them using the names on the motor housings under the RBS chamber. The "x", or lateral, axis code is 0001 and the "z", or vertical, axis code is 0010.

## Appendix II. Detailed Steps for Analyzing Two Dimensional PIXE Data

When the microprobe instrument is run on the Macintosh computer, the program stores information about each point in the scan rectangle in a data folder along with information about calibrated samples or standards. Each file in this folder is stored as a compressed Kmax™ histogram file.

When the microprobe instrument is finished taking data, it is necessary to move the information from the Macintosh computer, where Kmax™ runs, to the MS DOS machine where the GUPIX data fitting program resides. To accomplish this, the first runs a program written in True Basic that converts the files in the data folder into a format that GUPIX can read. Then the files are transferred to the MS DOS computer, either on 1.44M floppy disks or over ethernet, and stored in a subdirectory of the GUPIX directory. Once the files are in this subdirectory, a batch file is constructed so that GUPIX can be run in automatic mode to create a large file of concentration and position data that the user can display or further process with other programs .

To make a batch file, the directory command on the MS DOS machine is run with the "bare format" switch (/b). The output of this command is redirected to an ASCII file to generate a list containing the names of the files to be analyzed. This name list is needed so that the MS DOS computer can call each of the files sequentially into GUPIX. After this, the user will run GUPIX in non-batch mode on a representative spectrum, which will allow GUPIX to treat all of the other files in the data set in a similar fashion during batch mode. The user will then start the Word Perfect text editor so that the batch commands to GUPIX can be inserted in the ASCII text file list of all of the files in the data set that was generated earlier. Since the batch commands for each individual file are the same, the Word Perfect 5.1 search and replace function can be used to perform this

task many times automatically. Once the batch file is created, the MS DOS computer can run GUPIX with very minimal user interaction.

A. Converting Kmax™ packed data files to GUPIX93 format

1. The program CONVERT.TRU is invoked to convert the Kmax™ histogram files to a format that is readable by GUPIX on the MS DOS machine. CONVERT.TRU is a True Basic program that allows the user to specify the directory containing the files to be converted and the target directory where the converted files should be stored. The latter is usually a temporary folder on the desktop.

2. After the files have been converted, they are transferred to the MS DOS machine on 1.4M floppy disks or over the ethernet. The converted files are stored in a single subdirectory on the MS DOS machine so that they can be processed as a group. Once this has been accomplished, the files are ready to be fit by GUPIX.

B. Running GUPIX93 on an individual spectrum.

1. Before the files in the directory can be fit automatically in batch mode, information must be gleaned from them and supplied to GUPIX. This information is the "effective" detector solid angle (the solid angle that the detector apparently subtends at the target) and the energy calibration (the relationship between channel number in the data file and the energy of the X ray that would cause a peak in that channel).

2. First, an initial estimate of the energy calibration is obtained by identifying several characteristic peaks and their channel numbers from a standards spectrum. These data are fit to a linear function. The fit parameters, along with other information about X-ray filters and accumulated charge, are fed into GUPIX and GUPIX provides an output of the energy calibration that fits the data best, as well as estimates of concentrations of the various elements.

3. Since the concentrations of the various elements for the standards are known with high precision, the results from GUPIX are then compared with the standards data. The effective detector solid angle is varied until the concentrations yielded by GUPIX are acceptably close to the known concentrations.

C. Preparing to run GUPIX in batch mode.

1. When the files are transferred from the Macintosh computer, their names are not in a standard format. The file names must be modified by the user to conform to an easily recognizable format that includes date that data was taken and position in the scan rectangle that the particular histogram represents. This is accomplished with the DOS rename command. The format is: month (2 characters) day (2 characters) x position (2 characters) y position (2 characters). An example of the DOS command that would rename the raw files from the Macintosh to indicate that the data was taken on 2 FEB 95 is:

RENAME *.* 0202*.*

2. An ASCII text file is then made by using the directory command with the bare format switch toggled. This result is a name list that forms the beginning of the batch file that is eventually used by GUPIX to automatically fit the data.

DIR *.ASC /B > 02FEB.BAT

3. After this file is made, the Word Perfect 5.1 text editor is invoked to modify the file. Once the file is called in, the filenames are modified to conform to the format that GUPIX must use to call the files individually from the directory that it runs in. The data is stored in a subdirectory of the GUPIX directory

D. Sorting the GUPIX output file "conc.dat"

When GUPIX runs in batch mode, instead of directing the concentration data to the screen, it directs it to a file called "conc.dat" in the GUPIX home directory. This file contains all information about concentrations and uncertainties. A FORTRAN program "SORT-CONC.FOR" then strips off formatted text characters to make a large data array that contains position and concentration information. The result is in a comma-separated-variable (CSV) format that may be readily incorporated into a variety of programs, including spreadsheets and plotting programs.

## E. Source Code for PROCESS-SPECTRA.TRU

This program reads packed Kmax™ histogram files and converts them to one of several formats for further processing by spectral analysis programs. One such program is GUPIX93, which computes elemental concentrations from the PIXE spectra.

```
1000 OPTION NOLET
1010 REM
1020 REM                    ProcessSpectra
1030 REM
1040 REM  Original 07Oct93 JRV, USNA
1050 REM  Modified 21Oct93 JRV automatic conversions
1060 REM           11Nov93 JRV rds KMAX format w/ info in upper chan
1070 REM
1080 REM   file #1    FLIST
1090 REM   file #2   Kmax spectrum
1100 REM   file #3   output spectrum for GUPIX
1110 CLEAR
1120 PRINT "The KMAX files should have been saved with"
1130 PRINT "          Save |> Histograms As..."
1140 PRINT " "
1150 PRINT "The conversion process will proceed much easier"
1160 PRINT "  if all data files are placed in one sub-folder"
1170 PRINT " "
1180 PRINT "Do you wish to Continue ? [y/n]";
1190 INPUT ans$
1200 IF (ans$="n") or (ans$="N") then STOP
1210 PRINT " "
1220 PRINT "................................................."
1230 PRINT " "
1240 ASK DIRECTORY origin$
1250 PRINT "      Enter name of sub-folder         ";
1260 INPUT subdir$
1270 LET subdir$ = subdir$ & ":"
1280 LET inputdir$ = origin$ & subdir$
1290 PRINT "Enter DESTINATION DISK name for spectra";
1300 INPUT dest$
1310 LET dest$ = dest$ & ":"
1320 REM
1330 LET list$ = inputdir$ & "FLIST"
1340 OPEN #1: name list$, org text, create old
1350 PRINT " "
1360 PRINT "Choose Output File Format   [1=GUPIX, 2=Hypermet, 3=RUMP, 4=PCA]";
1370 INPUT format
1380 REM IF format<1 then GOTO 1360
1390 REM IF format>2 then GOTO 1360
1400 PRINT " "
1410 PRINT "................................................."
1420 PRINT " "
1430 LET ok=0
1440 FOR I=1 TO 300
1450     INPUT #1: str$
1460     LET str$=trim$(str$)
1470     LET str$=str$ & " "
1480     LET nchar=len(str$)
1490     LET start=1
1500     DO while start<nchar
1510        LET p=pos(str$," ",1)
1520        LET fname$=str$[1:p-1]
1530        IF fname$ = "Ok." then
1540           LET ok=ok+1
1550           IF ok=2 then
1560              PRINT " "
1570              PRINT "FILE CONVERSIONS COMPLETED"
1580              STOP
1590           END IF
1600           GO TO 1880
1610        ELSE
1620           IF fname$ = "FLIST" then GOTO 1820
1630           LET tmp$="Do you want the file " & fname$ & " Converted ? [y/n]"
```

```
1640            PRINT tmp$;
1650            INPUT ans$
1660            IF (ans$="y") or (ans$="Y") then
1670               SELECT CASE format
1680               CASE 1
1690                    CALL GUPIX(inputdir$,fname$,dest$)
1700               CASE 2
1710                    CALL LSI(inputdir$,fname$,dest$)
1720               CASE 3
1730                    CALL RUMP(inputdir$,fname$,dest$)
1740               CASE 4
1750                    CALL PCA(inputdir$,fname$,dest$)
1760               END SELECT
1770            END IF
1780            IF (ans$="n") or (ans$="N") then
1790               PRINT "NO CONVERSION"
1800            END IF
1810         END IF
1820         LET tmp$=str$
1830         LET str$=" "
1840         LET str$=tmp$[p+1:nchar]
1850         LET str$=ltrim$(str$)
1860         LET nchar=len(str$)
1870      LOOP
1880 NEXT I
1890 CLOSE #1
1900 END
1910
1920 SUB GUPIX(inputdir$,fname$,dest$)
1930      DIM spectrum(8192)
1940      LET finput$ = inputdir$ & fname$
1950      OPEN #2: name finput$, organization byte, create old, access input
1951      LET p=pos(fname$,".",1)
1952      LET nchar=len(fname$)
1953      IF p=0 then p=nchar+1
1954      LET fname$ = fname$[1:p-1]
1960      LET fout$ = dest$ & fname$ & ".asc"
1970      OPEN #3: name fout$, organization text, create new, access output
1980      SET #2: RECORD 513        ! skip Kmax header
1990      LET channels = 1024
2000      LET option = 1
2010      SET #2: RECSIZE 1
2020      PRINT #3:channels,option
2021      totcnt = 0
2030      FOR i = 1 TO channels
2040          READ #2: byte1$
2050          READ #2: byte2$
2060          READ #2: byte3$
2070          READ #2: byte4$
2080          LET byte1=ord(byte1$)
2090          LET byte2=ord(byte2$)
2100          LET byte3=ord(byte3$)
2110          LET byte4=ord(byte4$)
2120          LET spectrum(i)=byte1*16777216 + byte2*65536 + byte3*256 + byte4
2130          IF i<1000 then LET totcnt = totcnt + spectrum(i)
2140      NEXT i
2150      LET month = INT(spectrum(1011+1)/1000000)
2160      LET day = INT(spectrum(1011+1)/10000) - month*100
2170      LET year = 10000*FP(spectrum(1011+1)/10000)
2180      PRINT ,month,day,year
2190      PRINT #3:year
2200      IF month>=1 then LET offset=0
2210      IF month>=2 then LET offset=offset+31
2220      IF month>=3 then LET offset=offset+28
```

```
2230      IF month>=4 then LET offset=offset+30
2240      IF month>=5 then LET offset=offset+31
2250      IF month>=6 then LET offset=offset+30
2260      IF month>=7 then LET offset=offset+31
2270      IF month>=8 then LET offset=offset+30
2280      IF month>=9 then LET offset=offset+31
2290      IF month>=10 then LET offset=offset+30
2300      IF month>=11 then LET offset=offset+31
2310      LET leap=MOD(year,4)
2320      IF leap=0 then LET offset=offset+1
2330      LET doy = day + offset
2340      PRINT #3: doy
2350      LET start = spectrum(1012+1)  ! seconds past midnight
2360      PRINT ,"Start sec past midnight = ",start
2370      PRINT #3: spm
2380      LET finish = spectrum(1013+1)
2390      LET elapsed = finish-start
2400      DO while elapsed<0
2410         LET elapsed=elapsed+86400  ! assumes run no more than 24 hrs
2420      LOOP
2430      PRINT ,"Elapsed Time = ",elapsed
2431      LET livetime = spectrum(1014+1)
2432      PRINT ,"  Live Time  = ",livetime
2440      PRINT #3:livetime
2450 REM      LET totcnt=0              ! not sure what this is good for
2460      PRINT #3:totcnt
2470      LET label$ = ""
2480      FOR i = 1001 TO 1010
2490          LET p1 = MOD(spectrum(i),256)
2500          LET p2 = INT(spectrum(i)/256)
2510          LET p3 = INT(spectrum(i)/65536)
2520          LET p4 = INT(spectrum(i)/16777216)
2530          LET p2 = p2 - 65536*p3
2540          LET p3 = p3 - 16777216*p4
2550          LET label$ = label$ & CHR$(p1) & CHR$(p2) & CHR$(p3) & CHR$(p4)
2560          LET spectrum(i) = 0
2570      NEXT i
2580      PRINT ,label$
2590      PRINT " "
2600      PRINT #3:label$,"                                        "
2610      FOR j = 1 TO 1024
2620          PRINT #3: spectrum(j)
2630      NEXT j
2640      CLOSE #2
2650      CLOSE #3
2660 END SUB
2670
2680 SUB LSI(inputdir$,fname$,dest$)
2690      DIM spectrum(8192)
2700      LET finput$ = inputdir$ & fname$
2710      OPEN #2: name finput$, organization byte, create old, access input
2711      LET p=pos(fname$,".",1)
2712      LET nchar=len(fname$)
2713      IF p=0 then p=nchar+1
2714      LET fname$ = fname$[1:p-1]
2720      LET fout$ = dest$ & fname$ & ".lsi"
2730      OPEN #3: name fout$, organization text, create new, access output
2740      SET #3: MARGIN 100
2750      SET #2: RECORD 513        ! skip Kmax header
2760      LET channels = 8192
2770      LET option = 1
2780      SET #2: RECSIZE 1
2790      FOR i = 1 TO channels
2800          READ #2: byte1$
```

```
2810        READ #2: byte2$
2820        READ #2: byte3$
2830        READ #2: byte4$
2840        LET byte1=ord(byte1$)
2850        LET byte2=ord(byte2$)
2860        LET byte3=ord(byte3$)
2870        LET byte4=ord(byte4$)
2880        LET spectrum(i)=byte1*16777216 + byte2*65536 + byte3*256 + byte4
2890        REM       IF i>1000 then PRINT i,spectrum(i)
2900    NEXT i
2910    LET month = INT(spectrum(8161+1)/1000000)
2920    LET day = INT(spectrum(8161+1)/10000) - month*100
2930    LET year = 10000*FP(spectrum(8161+1)/10000)
2940    PRINT ,month,day,year
2950    IF month>=1 then LET offset=0
2960    IF month>=2 then LET offset=offset+31
2970    IF month>=3 then LET offset=offset+28
2980    IF month>=4 then LET offset=offset+30
2990    IF month>=5 then LET offset=offset+31
3000    IF month>=6 then LET offset=offset+30
3010    IF month>=7 then LET offset=offset+31
3020    IF month>=8 then LET offset=offset+30
3030    IF month>=9 then LET offset=offset+31
3040    IF month>=10 then LET offset=offset+30
3050    IF month>=11 then LET offset=offset+31
3060    LET leap=MOD(year,4)
3070    IF leap=0 then LET offset=offset+1
3080    LET doy = day + offset
3090    LET start = spectrum(8162+1)  ! seconds past midnight
3100    PRINT ,"Start sec past midnight = ",start
3110    LET finish = spectrum(8163+1)
3120    LET elapsed = finish-start
3130    DO while elapsed<0
3140       LET elapsed=elapsed+86400  ! assumes run no more than 24 hrs
3150    LOOP
3160    PRINT ,"Elapsed Time = ",elapsed
3161    LET livetime = spectrum(1014+1)
3162    PRINT ,"  Live Time  = ",livetime
3170    LET totcnt=0              ! not sure what this is good for
3180    LET label$ = ""
3190    FOR i = 8151 TO 8160
3200        LET p1 = MOD(spectrum(i),256)
3210        LET p2 = INT(spectrum(i)/256)
3220        LET p3 = INT(spectrum(i)/65536)
3230        LET p4 = INT(spectrum(i)/16777216)
3240        LET p2 = p2 - 65536*p3
3250        LET p3 = p3 - 16777216*p4
3260        LET label$ = label$ & CHR$(p1) & CHR$(p2) & CHR$(p3) & CHR$(p4)
3270        LET spectrum(i) = 0
3280    NEXT i
3290    PRINT ,label$
3300    PRINT " "
3310    PRINT #3:label$
3320    PRINT #3, USING "#####":" SIZE"
3330    SET #3: ZONEWIDTH 10
3340    LET form$ = "######### "
3350    FOR j = 1 TO 8192
3360        PRINT #3, USING form$: spectrum(j);
3370    NEXT j
3380    REM   PRINT #3: " "," "
3390    REM   PRINT #3, USING "#####":" SIZE"
3400    REM   FOR j = 2049 TO 4096
3410    REM       PRINT #3, USING form$: spectrum(j);
3420    REM   NEXT j
```

```
3430     REM   PRINT #3: " "," "
3440     REM   PRINT #3, USING "#####":" SIZE"
3450     REM   FOR j = 4097 TO 6144
3460     REM       PRINT #3, USING form$: spectrum(j);
3470     REM   NEXT j
3480     REM   PRINT #3: " "," "
3490     REM   PRINT #3, USING "#####":" SIZE"
3500     REM   FOR j = 6145 TO 8192
3510     REM       PRINT #3, USING form$: spectrum(j);
3520     REM   NEXT j
3530     REM   PRINT #3: " "," "
3540     CLOSE #2
3550     CLOSE #3
3560 END SUB
3570
3580 SUB RUMP(inputdir$,fname$,dest$)
3590     DIM spectrum(8192)
3600     LET finput$ = inputdir$ & fname$
3610     OPEN #2: name finput$, organization byte, create old, access input
3611     LET p=pos(fname$,".",1)
3612     LET nchar=len(fname$)
3613     IF p=0 then p=nchar+1
3614     LET fname$ = fname$[1:p-1]
3620     LET fout$ = dest$ & fname$ & ".asc"
3630     OPEN #3: name fout$, organization text, create new, access output
3640     SET #2: RECORD 513       ! skip Kmax header
3650     LET channels = 1024
3660     LET option = 1
3670     SET #2: RECSIZE 1
3680     FOR i = 1 TO channels
3690         READ #2: byte1$
3700         READ #2: byte2$
3710         READ #2: byte3$
3720         READ #2: byte4$
3730         LET byte1=ord(byte1$)
3740         LET byte2=ord(byte2$)
3750         LET byte3=ord(byte3$)
3760         LET byte4=ord(byte4$)
3770         LET spectrum(i)=byte1*16777216 + byte2*65536 + byte3*256 + byte4
3780     NEXT i
3790     REM       IF i>1000 then PRINT i,spectrum(i)
3800     PRINT #3:"EMPTY SWALLOW",CHR$(10)
3810     FOR j = 1 TO 1024
3820         PRINT #3: spectrum(j),CHR$(10)
3830     NEXT j
3840     CLOSE #2
3850     CLOSE #3
3860 END SUB
3870
3880 SUB PCA(inputdir$,fname$,dest$)
3890     DIM idata(33280)         ! 512 + 4*8192
3900     DIM spectrum(8192)
3910     LET finput$ = inputdir$ & fname$
3920     OPEN #2: name finput$, organization byte, create old, access input
3921     LET p=pos(fname$,".",1)
3922     LET nchar=len(fname$)
3923     IF p=0 then p=nchar+1
3924     LET fname$ = fname$[1:p-1]
3930     LET fout$ = dest$ & fname$ & ".pca"
3940     OPEN #3: name fout$, organization byte, create new, access output
3950     SET #2: RECORD 513       ! skip Kmax header
3960     LET channels = 1024
3970     LET option = 1
3980     SET #2: RECSIZE 1
```

```
3990     FOR X=1 TO 33280
4000         LET idata(X) = 0
4010     NEXT X
4020     LET elements = 512 + 4*channels
4030     REM read spectrum data points and load binary array lsb->msb
4040     FOR i = 0 TO 1023
4050         LET loc=4*i+513
4060         READ #2: byte1$
4070         READ #2: byte2$
4080         READ #2: byte3$
4090         READ #2: byte4$
4100         LET byte1=ORD(byte1$)
4110         LET byte2=ORD(byte2$)
4120         LET byte3=ORD(byte3$)
4130         LET byte4=ORD(byte4$)
4140         LET idata(loc+1)=ORD(byte4$)
4150         LET idata(loc+2)=ORD(byte3$)
4160         LET idata(loc+3)=ORD(byte2$)
4170         LET idata(loc+4)=0
4180         LET spectrum(i+1)=byte1*16777216 + byte2*65536 + byte3*256 + byte4
4190         IF i<1000 then   LET totcnt = totcnt+spectrum(i+1)
4200     NEXT i
4210     REM   gather scalar information
4220     LET month = INT(spectrum(1011+1)/1000000)
4230     LET day = INT(spectrum(1011+1)/10000) - month*100
4240     LET year = 10000*FP(spectrum(1011+1)/10000)
4250     PRINT ,"MDY",month,day,year
4260     PRINT #3:year
4270     IF month>=1 then LET offset=0
4280     IF month>=2 then LET offset=offset+31
4290     IF month>=3 then LET offset=offset+28
4300     IF month>=4 then LET offset=offset+30
4310     IF month>=5 then LET offset=offset+31
4320     IF month>=6 then LET offset=offset+30
4330     IF month>=7 then LET offset=offset+31
4340     IF month>=8 then LET offset=offset+30
4350     IF month>=9 then LET offset=offset+31
4360     IF month>=10 then LET offset=offset+30
4370     IF month>=11 then LET offset=offset+31
4380     LET leap=MOD(year,4)
4390     IF leap=0 then LET offset=offset+1
4400     LET doy = day + offset
4410     LET start = spectrum(1012+1)  ! seconds past midnight
4420     PRINT ,"Start sec past midnight = ",start
4430     LET finish = spectrum(1013+1)
4440     LET elapsed = finish-start
4441     PRINT ,"Elapsed Time [sec] = ",elapsed
4442     LET livetime = spectrum(1014+1)
4443     PRINT ,"  Live Time  = ",livetime
4445     PRINT ,"Total Counts in Spectrum = ",totcnt
4450     LET digit1 = INT(livetime/1000000)
4460     LET digit2 = MOD( INT(livetime/10000) , 10 )
4470     LET digit3 = MOD( INT(livetime/1000) , 10 )
4480     LET digit4 = MOD( INT(livetime/100) , 10 )
4490     LET digit5 = MOD( INT(livetime/10) , 10 )
4500     LET digit6 = MOD( livetime, 10 )
4510     LET idata(0+1) = 16*digit1 + digit2
4520     LET idata(1+1) = 16*digit3 + digit4
4530     LET idata(2+1) = 16*digit5 + digit6
4540     LET idata(3+1) = 255     ! 0FFh
4550     LET idata(4+1) = 0
4560     IF channels=1024 then LET idata(5+1) = 3
4570     IF channels=8192 then LET idata(5+1) = 0
4580     LET idata(6+1) = 0
```

```
4581     blank2$ = "  "
4590     FOR loc = 07 TO 21
4600         LET idata(loc+1) = 32 !ORD(blank2$)
4610     NEXT loc
4620     FOR loc = 22 TO 33
4630         LET idata(loc+1) = 32 !ORD(blank2$)
4640     NEXT loc
4650     FOR loc = 34 TO 45
4660         LET idata(loc+1) = 32 !ORD(blank2$)
4670     NEXT loc
4680     LET idata(257+1) = 255   ! 0FFh
4690     LET idata(258+1) = 0
4700     LET Hbyte = INT(channels/256)
4710     LET Lbyte = MOD( channels,256 )
4720     LET idata(266+1) = Lbyte
4730     LET idata(267+1) = Hbyte
4740     REM     LET label$ = ""
4750     REM     FOR i = 1001 TO 1010
4760     REM         LET p1 = MOD(spectrum(i),256)
4770     REM         LET p2 = INT(spectrum(i)/256)
4780     REM         LET p3 = INT(spectrum(i)/65536)
4790     REM         LET p4 = INT(spectrum(i)/16777216)
4800     REM         LET p2 = p2 - 65536*p3
4810     REM         LET p3 = p3 - 16777216*p4
4820     REM         LET label$ = label$ & CHR$(p1) & CHR$(p2) & CHR$(p3) & CHR$(p4)
4830     REM         LET spectrum(i) = 0
4840     REM     NEXT i
4850     REM     PRINT ,label$
4860     REM     PRINT " "
4870     REM     PRINT #3:label$
4880     REM
4890     REM  print the filled binary array
4900     REM
4910     FOR j = 1 TO elements
4920         PRINT #3: CHR$(idata(j))
4930     NEXT j
4940     CLOSE #2
4950     CLOSE #3
4960 END SUB
```

## F. Source Code for SORT-CONC.FOR

This program reads CONC.DAT files made by GUPIX93, performs H(Z) corrections referenced to iron, and sorts them into a table with the format

hpos, vpos, (conc(iz), iz = zmin,zmax),

where hpos and vpos are the horizontal and vertical positions, respectively, and conc(iz) is the concentration at (hpos, vpos) of the element with atomic number Z.

This table is written into the output file CTABLE.CSV, which is in a comma-separated-variable-format that can be read by many other applications, including spreadsheets and plotting programs.

```
c
c   SORT-CONC
c
c   Reads CONC.DAT files made by GUPIX93,
c   performs H(Z) corrections referenced to Iron,
c   and then sorts them into a table with the format:
c       hpos,vpos,(conc(iz),iz=zmin,zmax)
c   The resulting output file CTABLE.CSV is in the
c    comma-separated-variable format that can be
c    directly read by all spreadsheet-type applications.
c
c   The statistics of whether GUPIX93 thinks the element
c   is present or not is kept track of by the array
c   nexist(iz).  Each "Y" increases nexist by 1, while
c   a "N" decreases nexist by 1.  The average concentration
c   is also kept track of in aveconc(iz)
c
      character*1 flag,exist(40)
      character*2 hpos,vpos
      character*4 dummy
      character*5 queue
      character*20 finput
      integer zmin,zmax,nexist(40)
      real lod,hfactor(40),conc(40),uconc(40),HFe,Hz
      real aveconc(40),aveunc(40),avelod(40)
      data exist/40*' '/
      data nexist/40*0/
      data aveconc/40*0.0/
      data aveunc/40*0.0/
      data avelod/40*0.0/
      nfile=0
      zmax=-99
      zmin= 99
      HFe=2.5299e-5     ! original H factor for Iron
c
      write(*,99)
   99 format(' Input conc file :: ',$)
      read(*,'(a20)') finput
      open(unit=1,file=finput,status='old')
      open(unit=10,file='ctable.CSV',status='new',recl=400)
      do 50 i=1,10000
        read(1,100,end=9999) queue,hpos,vpos
  100   format(a5,16x,a2,a2,/)
        if(queue.ne.' File') go to 50
        nfile=nfile+1
        read(1,'(a4,/)') dummy
        read(1,'(a4,/)') dummy
        read(1,'(a4,/)') dummy
        do 10 j=1,30
          read(1,200,err=11,end=9999) iz,tempc,tempu,lod,flag
  200     format(2x,i2,25x,3f10.1,2x,a1,/)
          z=log(float(iz))
          Hz=exp( 12.056-19.943*z+3.9768*z*z )
          zmin=min(zmin,iz)
          zmax=max(zmax,iz)
          conc(iz)=tempc*HFe/Hz
          uconc(iz)=tempu*HFe/Hz
          aveconc(iz)=aveconc(iz)+conc(iz)
          aveunc(iz)=aveunc(iz)+uconc(iz)*uconc(iz)
          avelod(iz)=avelod(iz)+lod*HFe/Hz
          exist(iz)=flag
          if(flag.eq.'Y') nexist(iz)=nexist(iz)+1
          if(flag.eq.'N') nexist(iz)=nexist(iz)-1
   10   continue
```

```
c        write(*,*) ' zmin, zmax =',zmin,zmax
   11    write(10,300) hpos,vpos,(conc(k),k=zmin,zmax)
         write( *,301) nfile,hpos,vpos,(exist(k),k=zmin,zmax)
  300    format(1x,a2,',',a2,30(',',f10.1))    ! CSV format
  301    format(1x,i4,1x,a2,a2,1x,40a1)
         backspace(1)
   50 continue
 9999 continue
c
c     Print out existance stats
c
      write(*,400) nfile
  400 format(' There were ',i3,' scan locations')
      write(*,401)
  401 format(1x,' Z    Occurances   Ave Conc [ppm]',
     * '   Uncertainty      Ave LOD')
  402 format(1x,i2,7x,i4,9x,f10.1,4x,f10.1,4x,f10.1)
      do 60 k=zmin,zmax
        temp1=aveconc(k)/nfile
        temp2=sqrt(aveunc(k))/nfile
        temp3=avelod(k)/nfile
        write(*,402) k,nexist(k),temp1,temp2,temp3
   60 continue
      stop
      end
```

## G. Source Code for CORRELATE.FOR

This program reads the summary CTABLE.CSV written by SORT-CONC.FOR and creates two dimensional plots of the concentration of element A versus element B. Elements to be plotted are specified when the program is run.

An optional file CTABLE.PIC may be supplied which specifies different plotting symbols for different regions of the scan area. This may be useful for distinguishing between positions inside and outside an inclusion.

```
c
c   CORRELATE.for
c
c   Reads CTABLE.OUT summary prepared on PC which
c   is intended for SPYGLASS/TRANSFORM and makes
c   plots of ELEMENT A vs ELEMENT B; for all A and B.
c
c   If a file may also be created which specifies the
c   plotting character to be used at each point: CTABLE.PIC
c   This is useful for identifying any clustering phenomena
c   occuring.
c
      integer hpos(400),vpos(400)
      real conc(40,400)
      real csum(40),xconc(400),yconc(400)
c
      integer hmin,hmax,vmin,vmax,pic(20,20)
c
      character*2 zlabel
      character*6 xlabel,ylabel
      character*20 filecsv,filepic
      data csum/40*0.0/
      data pic/400*1.0/
c
c Read CSV formatted Concentration Table
c
      write(*,1)
    1 format(' Filename of CSV formatted concentration table : ',$)
      read(*,'(a20)') filecsv
      open(unit=1,file=filecsv,status='old')
      write(*,3)
    3 format(' Enter Zmin, Zmax for that file [int] : ',$)
      read(*,*) izmin,izmax
      do 15 i=1,400
        read(1,*,end=16) hpos(i),vpos(i),(conc(iz,i),iz=izmin,izmax)
        do 10 iz=izmin,izmax
          csum(iz)=csum(iz)+conc(iz,i)
   10   continue
   15 continue
   16 nscan=i-1
c
c Read Picture Table
c
      write(*,100)
  100 format(' Is there a picture/symbol file ? [cr=no] << ',$)
      read(*,'(a1)') flag
      if(flag.eq.' ') go to 30
       write(*,2)
    2  format(' Enter filename of PIC file : ',$)
       read(*,'(a20)') filepic
       open(unit=2,file=filepic,status='old')
       read(2,'(a1)') dummy
       read(2,*) hmin,hmax
       read(2,*) vmax,vmin
       read(2,'(a1)') dummy
       do 22 v=vmax,vmin,-1
         read(2,'(2x,20i1)') (pic(h+1,v+1),h=hmin,hmax)
c         write(*,'(1x,20i1)')  (pic(h+1,v+1),h=hmin,hmax)
   22  continue
   30 continue
c
c Plot section
c
      write(*,4)
```

```
    4 format(/,' Single plot, or all plots? [1=one] : ',$)
      read(*,'(a1)') ans
      if(ans.ne.'1') go to 99
c
c     Individual plots
c
      write(*,5)
    5 format(' Enter Z1(y), Z2(x) : ',$)
      read(*,*) iza,izb
      call pgbegin(0,'?',1,1)
      call pgslw(2)
      ymax=0.0
      do 32 i=1,nscan
        yconc(i)=conc(iza,i)
        ymax=max(ymax,yconc(i))
   32 continue
      xmax=0.0
      do 42 i=1,nscan
        xconc(i)=conc(izb,i)
        xmax=max(xmax,xconc(i))
   42 continue
      call pgask(.false.)
      call pgadvance
      call pgvport(0.2,0.8,0.3,0.7)
      call pgwindow(0.,xmax,0.,ymax)
      call pgsch(0.8)
      call pgbox('abcnt',0.0,0,'abcntv',0.0,0)
      call pgsch(1.5)
      if(flag.ne.' ') then
        do 44 i=1,nscan
          isym=pic(hpos(i)+1,vpos(i)+1)
          call pgpoint(1,xconc(i),yconc(i),isym)
   44   continue
      else
        call pgpoint(nscan,xconc(1),yconc(1),1)
      endif
      encode(2,'(i2)',zlabel) iza
      ylabel = 'Z = ' // zlabel
      encode(2,'(i2)',zlabel) izb
      xlabel = 'Z = ' // zlabel
      call pgsch(2.0)
      call pglabel(xlabel,ylabel,' ')
   40 continue
      call pgupdt
      call pgend
      read(*,'(a1)') ans
      go to 8888
c
c     Free running plot section of Z1 x Z2
c
   99 continue
      write(*,6)
    6 format(' Enter cutoff for plot exclusion [ppm] : ',$)
      read(*,*) cutoff
      do 150 iza=izmin,izmax
        if( (csum(iza)/float(nscan)).lt.cutoff ) go to 150
        call pgbegin(0,'?',3,6)
        ymax=0.0
        do 132 i=1,nscan
          yconc(i)=conc(iza,i)
          ymax=max(ymax,yconc(i))
  132   continue
        do 140 izb=izmin,izmax
          if( (csum(izb)/float(nscan)).lt.cutoff ) go to 140
```

```
          xmax=0.0
          do 142 i=1,nscan
            xconc(i)=conc(izb,i)
            xmax=max(xmax,xconc(i))
  142     continue

          call pgask(.false.)
          call pgadvance
          call pgvport(0.2,0.8,0.2,0.8)
          call pgwindow(0.,xmax,0.,ymax)
          call pgsch(1.5)
          call pgbox('abcnt',0.0,0,'abcntv',0.0,0)
          if(flag.ne.' ') then
             do 144 i=1,nscan
               isym=pic(hpos(i)+1,vpos(i)+1)
               call pgpoint(1,xconc(i),yconc(i),isym)
  144        continue
          else
             call pgpoint(nscan,xconc(i),yconc(i),1)
          endif
c
          encode(2,'(i2)',zlabel) iza
          ylabel = 'Z = ' // zlabel
          encode(2,'(i2)',zlabel) izb
          xlabel = 'Z = ' // zlabel
          call pgsch(2.0)
          call pglabel(xlabel,ylabel,' ')
c          if(izb.eq.4) go to 8888          ! for testing purposes

  140   continue
        call pgupdt
        call pgend
c        if(izb.ne.0) go to 8888  ! for testing purposes
  150 continue
c
c 8888 call pgupdt
c      call pgend
 8888 stop
      end
```